

A STUDY OF THE EFFECT OF TIME
AND TEMPERATURE OF PRESOAKING AND THE TEMPERATURE OF
ACETYLTATION ON THE RATE AND DEGREE OF THE PARTIAL
ACETYLTATION OF DIFFERENT VARIETIES OF COTTON FIBERS

A THESIS

Presented to
the Faculty of the Graduate Division

by

Emmet Dennis Owens

In Partial Fulfillment
of the Requirement for the Degree
Master of Science in Textile Engineering

Georgia Institute of Technology

June 1953

THESIS
096

Library
U. S. Naval Postgraduate School
Monterey, California



"In presenting the dissertation as a partial fulfillment of the requirements for an advanced degree from the Georgia Institute of Technology, I agree that the Library of the Institution shall make it available for inspection and circulation in accordance with its regulations governing materials of this type. I agree that permission to copy from, or to publish from, this dissertation may be granted by the professor under whose direction it was written, or, in his absence, by the Dean of the Graduate Division when such copying or publication is solely for scholarly purposes and does not involve potential financial gain. It is understood that any copying from, or publication of, this dissertation which involves potential financial gain will not be allowed without written permission.

A STUDY OF THE EFFECT OF TIME
AND TEMPERATURE OF PRESOAKING AND THE TEMPERATURE OF
ACETYLATION ON THE RATE AND DEGREE OF THE PARTIAL
ACETYLATION OF DIFFERENT VARIETIES OF COTTON FIBERS

A THESIS

Presented to
the Faculty of the Graduate Division

by

Emmet Dennis Owens
"

In Partial Fulfillment
of the Requirement for the Degree
Master of Science in Textile Engineering

Georgia Institute of Technology

June 1953

A STUDY OF THE EFFECT OF TIME
AND TEMPERATURE OF PRESOAKING AND THE TEMPERATURE OF
ACETYLATION ON THE RATE AND DEGREE OF THE PARTIAL
ACETYLATION OF DIFFERENT VARIETIES OF COTTON FIBERS

Approved:

ACKNOWLEDGEMENTS

On the completion of this study, I wish to express my gratitude to Dr. James L. Taylor of the Georgia Institute of Technology for his invaluable assistance and guidance. I also wish to thank Professor C. A. Jones and Dr. Nathan Sugarman for their helpful advice.

I wish to thank the United States Navy for making the undertaking and completion of this study possible.

I would also like to extend my thanks to the Southern Regional Research Laboratory, New Orleans, La. for furnishing the cotton used in this study.

A STUDY OF THE EFFECT OF TIME
AND TEMPERATURE OF PRESOAKING AND THE TEMPERATURE OF
ACETYLATION ON THE RATE AND DEGREE OF THE PARTIAL
ACETYLATION OF DIFFERENT VARIETIES OF COTTON FIBERS

ABSTRACT

It was the purpose of this problem to determine (1) the effect of time and temperature of presoaking on the rate and degree of the partial acetylation of different varieties of cotton fibers with the time and temperature of the acetylation held constant and (2) to study the effect of the acetylation temperature under constant conditions of presoaking.

The degradation of cotton fibers in the production of partially acetylated cotton is due primarily to the time and temperature used in the acetylation process. It is therefore of prime importance to determine some method of reducing the time and temperature of acetylation to a minimum and still obtain the desired acetyl content. The effect of the time and temperature of presoaking on the rate and degree of acetylation was thus determined in order to obtain the optimum conditions of presoaking.

Six different varieties of cotton fibers representing a wide range of physical properties were selected for this study. The cottons selected were Memphis, Empire Bale 92, Bob Shaw, Stoneville 2B, Acala 1517 and Lockett 140.

THE JOURNAL OF THE ROYAL ANTHROPOLOGICAL INSTITUTE

1906

PUBLISHED BY THE
EDUCATION OFFICE, LONDON

THE JOURNAL OF THE
ROYAL ANTHROPOLOGICAL INSTITUTE

OF GREAT BRITAIN AND IRELAND

OF THE ANTHROPOLOGICAL INSTITUTE
OF GREAT BRITAIN AND IRELAND

OF THE ANTHROPOLOGICAL INSTITUTE
OF GREAT BRITAIN AND IRELAND

OF THE ANTHROPOLOGICAL INSTITUTE
OF GREAT BRITAIN AND IRELAND

OF THE ANTHROPOLOGICAL INSTITUTE
OF GREAT BRITAIN AND IRELAND

OF THE ANTHROPOLOGICAL INSTITUTE
OF GREAT BRITAIN AND IRELAND

OF THE ANTHROPOLOGICAL INSTITUTE
OF GREAT BRITAIN AND IRELAND

OF THE ANTHROPOLOGICAL INSTITUTE
OF GREAT BRITAIN AND IRELAND

OF THE ANTHROPOLOGICAL INSTITUTE
OF GREAT BRITAIN AND IRELAND

OF THE ANTHROPOLOGICAL INSTITUTE
OF GREAT BRITAIN AND IRELAND

OF THE ANTHROPOLOGICAL INSTITUTE
OF GREAT BRITAIN AND IRELAND

OF THE ANTHROPOLOGICAL INSTITUTE
OF GREAT BRITAIN AND IRELAND

OF THE ANTHROPOLOGICAL INSTITUTE
OF GREAT BRITAIN AND IRELAND

OF THE ANTHROPOLOGICAL INSTITUTE
OF GREAT BRITAIN AND IRELAND

OF THE ANTHROPOLOGICAL INSTITUTE
OF GREAT BRITAIN AND IRELAND

All acetylations in Part 1 of this study were carried out at 64° F. for 45 minutes. The time of presoaking was varied from 10 minutes to 240 minutes and the temperatures used were 70° F., 100° F., 130° F., and 170° F. In Part 2 all acetylations were carried out for 45 minutes at different temperatures ranging from 58° F. to 82° F. in six degree increments. The optimum presoaking conditions, 10 minutes at 170° F., were determined from Part 1 and used for all acetylations in Part 2.

All presoakings and acetylations were carried out in a one-pound package dyeing machine modified with cooling coils in such a manner that positive temperature control was attained at all times.

The acetylated samples were analyzed for acetyl content, moisture per cent, fiber fineness, fiber strength, and evenness of acetylation. The values of the acetyl contents were plotted against the times and temperatures of presoaking and the temperatures of acetylation and the graphs carefully analyzed.

From the results obtained it was concluded that an increase in the temperature of the glacial acetic acid used for presoaking increases the rate in which maximum acetylation is reached but it does not effect the maximum degree of acetylation obtained under constant acetylation conditions. It was found that different fibers acetylated at different rates and degrees and generally the more immature the fiber the greater the rate and degree of acetylation and conversely the more mature the fiber the less the rate and degree of acetylation under constant conditions of presoaking and acetylation. Finally, it was determined that

the higher the temperature used in acetylation, the greater the degree of acetylation and the greater the degradation of the cotton fibers.

TABLE OF CONTENTS

	Page
ACKNOWLEDGEMENT.	iii
ABSTRACT	iv
LIST OF TABLES	ix
LIST OF ILLUSTRATIONS.	xi
Chapter	
I. INTRODUCTION.	1
History	2
Statement of the Problem.	6
Importance of the Study	6
Method of Approach	7
II. THEORETICAL CONSIDERATIONS.	10
III. INSTRUMENTATION AND EQUIPMENT	18
IV. EXPERIMENTAL PROCEDURE.	21
Selection of Cotton Fibers.	21
Testing Prior to Acetylation.	21
Moisture Content - Brabender	21
Fiber Fineness - Micronaire.	21
Fiber Strength - Pressley.	22
Preparation of the Cotton Prior to Acetylation.	22
Conditioning	22
Prescaking	22

Chapter	Page
Acetylation.	23
Preparation of the Acetylation Mixture.	23
Acetylation	24
Treatment after Acetylation.	24
Washing	24
Drying.	25
Conditioning.	25
Testing After Acetylation.	25
Physical Properties	25
Analysis for Acetyl Content	25
Dye Test for Evenness of Acetylation.	25
V. DISCUSSION OF RESULTS.	27
VI. CONCLUSIONS.	52
VII. RECOMMENDATIONS.	54
BIBLIOGRAPHY.	55
APPENDIX.	59

LIST OF TABLES

Table	Page
1. Summary of Physical and Chemical Properties of the Original Cotton Fibers.	9
2. A Comparison of the Acetyl Content of Acala 1517 Cotton at Various Times and Temperatures of Presoaking	27
3. A Comparison of the Acetyl Content of Various Cotton Fibers Presoaked at 70° F. for Different Times.	29
4. Effect of the Temperature of Presoaking at 70° F. on the Degree of Acetylation.	33
5. Effect of the Temperature of Presoaking at 100° F. on the Degree of Acetylation.	33
6. Effect of the Temperature of Presoaking at 130° F. on the Degree of Acetylation.	34
7. Effect of the Temperature of Presoaking at 170° F. on the Degree of Acetylation.	34
8. Effect of Temperature of Acetylation on the Degree of Acetylation.	35
9. Per Cent Moisture Content of Acetylated Samples Presoaked at 70° F.	60
10. Per Cent Moisture Content of Acetylated Samples Presoaked at 100° F.	60
11. Per Cent Moisture Content of Acetylated Samples Presoaked at 130° F.	61
12. Per Cent Moisture Content of Acetylated Samples Presoaked at 170° F.	61
13. Per Cent Moisture Content of Samples Acetylated at Different Temperatures	62
14. Micronaire Readings (Fiber Fineness) of Acetylated Samples Presoaked at 70° F.	63
15. Micronaire Readings (Fiber Fineness) of Acetylated Samples Presoaked at 100° F.	63

Table	Page
16. Micronaire Readings (Fiber Fineness) of Acetylated Samples Presoaked at 130° F.	64
17. Micronaire Readings (Fiber Fineness) of Acetylated Samples Presoaked at 170° F.	64
18. Micronaire Readings (Fiber Fineness) of Samples Acetylated at Different Temperatures.	65
19. Pressley Index (Fiber Strength) of Acetylated Samples Presoaked at 70° F.	66
20. Pressley Index (Fiber Strength) of Acetylated Samples Presoaked at 100° F.	66
21. Pressley Index (Fiber Strength) of Acetylated Samples Presoaked at 130° F.	67
22. Pressley Index (Fiber Strength) of Acetylated Samples Presoaked at 170° F.	67
23. Pressley Index (Fiber Strength) of Samples Acetylated at Different Temperatures.	68

LIST OF ILLUSTRATIONS

Figure	Page
1. Front View of the Acetylation Machine.	19
2. Top View of the Acetylation Machine	20
3. The Change in Acetyl Content with Presoaking Time at 70° F.	36
4. The Change in Acetyl Content with Presoaking Time at 100° F.	37
5. The Change in Acetyl Content with Presoaking Time at 130° F.	38
6. The Change in Acetyl Content with Presoaking Time at 170° F.	39
7. The Change in Acetyl Content with Acetylation Temperature Using a 10 Minute, 170° F. Presoak	40
8. The Change in Acetyl Content with Presoaking Tem- perature Using a 10 Minute Presoaking Period	41
9. The Change in Acetyl Content with Presoaking Tem- perature Using a 30 Minute Presoaking Period	42
10. The Change in Acetyl Content with Presoaking Tem- perature Using a 60 Minute Presoaking Period	43
11. The Change in Acetyl Content with Presoaking Tem- perature Using a 120 Minute Presoaking Period.	44
12. The Change in Acetyl Content with Presoaking Tem- perature Using a 240 Minute Presoaking Period.	45
13. The Change in Acetyl Content with Presoaking Time for Memphis Cotton at Different Presoak Temperatures	46
14. The Change in Acetyl Content with Presoaking Time for Empire Bale 92 Cotton at Different Presoak Temperatures.	47
15. The Change in Acetyl Content with Presoaking Time for Bob Shaw Cotton at Different Presoak Temperatures.	48

Figure	Page
16. The Change in Acetyl Content with Presoaking Time for Stoneville 2B Cotton at Different Presoak Temperatures.	49
17. The Change in Acetyl Content with Presoaking Time for Acala 1517 Cotton at Different Presoak Temperatures	50
18. The Change in Acetyl Content with Presoaking Time for Lockett 140 Cotton at Different Presoak Temperatures.	51

CHAPTER I

INTRODUCTION

In recent years considerable attention has been focused on the use of partially acetylated cotton as a textile fiber. The chemical modification of cotton by partial acetylation imparts many new and valuable properties to the cotton fiber while at the same time its physical appearance and general textile qualities remain virtually unchanged. The new characteristics of the chemically modified cotton fibers include resistance to mildew and microbiological rotting; resistance to the effects of high temperature and continued heat; low moisture regain and high electrical resistance, as compared with untreated cotton; and resistance to coloring with many direct dyes.¹

Partially acetylated cotton appears to be unaltered physically both in the acetylating mixture and in the dried condition. It possesses the same tensile strength per unit weight of cellulose and the same elasticity as unacetylated cotton, and shows only a somewhat harder handle. The characteristic structure of the cotton fiber is preserved entirely; even the light reflections in polarized light are the same. The only noticeable difference is that low acetylated fibers appear to

¹Albert S. Cooper, and others, "Partial Acetylation of Cotton," Textile Industries, 116-1 (January, 1952), 97-102, 194-95.

be more rod-like than the original fibers and the fibers increased in weight by approximately 25 per cent without shrinkage in length.²

The degree of acetylation of the cotton fiber considered in this study is based on that of the monacetate, corresponding to approximately one-third of complete acetylation or about 21 per cent acetyl content. The partial acetylation of cotton is an example of esterification which consists in the introduction of acetyl groups throughout the cellulose to replace an equivalent part of its hydroxyl groups of which cotton contains about 30 per cent by weight.

History.--In 1901 two British chemists, C. F. Cross and E. J. Bevin, produced a low acetylated cotton fiber by treating bleached cotton with boiling acetic anhydride in the presence of distilled sodium acetate. Although possessing the properties of partially acetylated cotton; i.e. (a) it absorbed much less than the usual amount of hygroscopic moisture from the atmosphere and (b) it was unaffected by the usual solvents for cellulose acetates; these fibrous acetates were of no practical use since they had lost all tensile strength, probably due to the high reaction temperature.³

C. F. Cross and Briggs altered the original process in 1907 with a view to conducting the acetylation at low temperatures. In this process the cellulose was impregnated with a mixture of acetic anhydride, acetic acid, and a salt like condensing agent and then squeezed until a certain

²E. Chippindale, "Low Acetylation of Cellulose Fibers," Society of Dyers and Colourists, 1 (1934), 142-49.

³Ibid., p. 142.

increase in weight was obtained and afterwards acetylating by warming in a closed apparatus. This process was found to be of no commercial value due to the complicated apparatus required and to the fact that irregularly acetylated fibers were obtained due to lack of control during the reaction.⁴

Work continued on the partial acetylation of cotton, mainly in England, with the object of producing partially acetylated cotton which would retain its tensile strength after acetylation. In 1928 partially acetylated cotton yarns were the subject of E. P. 280,493 and were registered under the names of Cotopa (non-lustrous) and Crestol (lustrous).⁵

The usual acetylation procedure today is essentially the one that is described in A. C. Thaysen's British patents and intended particularly to impart resistance to mildew and rot.⁶

Extensive investigation has been carried out on partially acetylated cotton in order to determine more about the new properties imparted to the cotton fiber. The Department of Agriculture, through its Southern Regional Research Laboratory, New Orleans, La., has conducted extensive research in the production and uses of acetylated yarn and fabrics.

In 1920, C. Doree reported that cotton fabrics, acetylated to the monoacetate stage, immersed in sea water were completely unaffected after

⁴Ibid., p. 142.

⁵Ibid., p. 145.

⁶Charles Goldthwait, "Acetylation to Make a New Textile," Yearbook of Agriculture, 1950-51, p. 422.

sixteen weeks while unacetylated cotton was destroyed in three weeks.⁷ Thaysen and his coworkers found that partially acetylated cotton resisted rotting by soil micro-organisms when buried in the ground and reported on a partially acetylated cotton fabric which withstood such burial for seven years.⁸

Partially acetylated cotton has an advantage over goods treated with the usual mildew and rot-proofing agents because it is not discolored, odorous, sticky, nor poisonous.⁹ It has been found that bags made of partially acetylated cotton for holding zeolite in household water-softening systems remained intact during a normal years service, whereas ordinary bags developed holes in a month or two. Acetylated fish net twines tested in ocean water were found to last two to three times as long as twines treated with tar and eight to ten times as long as untreated twines. The nets of acetylated twines are more flexible and easier to handle than nets protected by tar.¹⁰ Tests on acetylated sandbags showed that after two years exposure to the weather the bags on the bottom of the pile retained 35 to 40 per cent of their breaking strength and those on the top retained 35 to 100 per cent of their breaking strength in contrast to complete deterioration of untreated bags in one-fourth the time.¹¹

⁷C. Doree, "The Action of Sea Water on Cotton and Other Textile Fibers," Biochemistry Journal, 14 (1920), p. 713.

⁸G. F. Goldthwait, J. McLaren and S. T. Voorhies, Jr., "Acetylated Cotton Highly Resistant to Rotting," Textile World, 96-1 (1946), p. 117.

⁹Ibid., p. 116

¹⁰Copper and others, op. cit., p. 98.

¹¹Walter Scott, "Some Recent Developments in Cotton Research at Southern Regional Research Laboratory," Textile Research Journal, 19 (1949), p. 440.

The resistance of partially acetylated cotton to high temperatures and continued heat have been extensively investigated. Tests were made on the comparison between acetylated cotton fabrics and unacetylated fabrics at 160° F. under different conditions of humidity, etc. and it was found that in all cases the acetylated fabrics were much more heat resistant. The tensile strength of the unacetylated cotton fabrics were greatly reduced while that of the acetylated cotton were only slightly affected.¹² A sheeting of partially acetylated cotton has been found to last four to five times as long as the usual cotton canvas covers for laundry "hot head presses". Examples of the effect of dry heat with full exposure to air in an ordinary drying oven indicated that the acetylated cotton would require more than seven times as long to become weakened to half strength at 320° F.¹³

Acetylated cotton has been used as tire cords with good results by the British for a number of years. It was found to be particularly effective in tropic climates.¹⁴ Acetylated cotton swells less when wet and it is of particular value when cotton textiles have to maintain their weight, length and width unchanged under varying weather conditions. Partially acetylated cotton is also said to bond better than ordinary

¹²E. Honold, J. Poynot and A. Cuculla, "Heat Resistance of Partially Acetylated Cotton Fabrics," Textile Research Journal, 22-1 (1952), 25-29.

¹³A. S. Cooper and others, op. cit., p. 97.

¹⁴C. M. Blow and W. J. Knight, "Textile Fibres and Rubber as Associated Materials for Manufacture," Textile Institute Journal Proceedings, 35 (1944), p. 12.

cotton with plastics, such as cellulose acetates, etc. Owing to its low hygroscopicity, about half of that of unacetylated cotton, it finds use as electrical insulation material.¹⁵ As more fundamental knowledge concerning partially acetylated cotton is discovered the uses of this cotton will greatly increase.

Statement of the Problem.---The partial acetylation of cotton fibers is carried out in three principal steps: (1) preparation of the cotton for acetylation, (2) the acetylation reaction and (3) washing and drying.

It was the purpose of this study to determine (1) the effect of the time and temperature of presoaking on the rate and degree of acetylation of different varieties of cotton fibers with the time and temperature of the acetylation held constant and (2) to study the effect of the acetylation temperature on the degree of acetylation under constant conditions of presoaking.

Importance of the Study.---At the present time partially acetylated cotton is being produced only on a limited scale in this country and although considerable research is being conducted, primarily by the Southern Regional Research Laboratory, New Orleans, La., there still remain many inherent fundamental physical and chemical aspects of the acetylation reaction to be determined. Relatively little research has been carried out on the raw stock itself and it is the purpose of this study to determine the relationship of the presoaking conditions, using glacial

¹⁵C. F. Goldthwait, op. cit., p. 421.

acetic acid as the presoaking reagent, to the rate and degree of acetylation. Previous acetylations of raw stock have usually been carried out using an overnight presoak at room temperatures. Acetylated cotton produced on a commercial scale must necessarily be a continuous process carried out within the shortest possible time with the least amount of damage to the fibers. Heuser¹⁶ has shown that the amount of degradation of cellulose depends upon the time and temperature of acetylation, independently of the acetyl values reached. It is therefore of prime importance to determine some method by which the time and temperature of acetylation could be reduced and still obtain the desired acetyl values. To that end, various conditions of time and temperature of presoaking and temperatures of acetylation were studied, the acetyl content determined and the results analyzed.

Method of Approach.--Six different varieties of raw cotton were selected for this study. These cottons were chosen to represent a wide range of physical properties such as maturity, fiber fineness and tensile strength as illustrated in Table 1.

In the first part of this study the cottons were acetylated under uniform conditions of time and temperature, i.e. 45 minutes at 64° F., with the time and temperature of presoaking as variables. The times used were 10, 30, 60, 120, and 240 minutes and the temperatures selected were 70° F., 100° F., 130° F., and 170° F.

In the second part of this study the time and temperature of presoaking was held constant at the optimum conditions of presoaking as

¹⁶Emil Heuser and others, "Acetylation of Cellulose in Phosphoric Acid Solution," Industrial and Engineering Chemistry, 40 (1948), 1500-06.

determined from Part I. These conditions were determined to be 10 minutes at 170° F. The acetylation temperature was varied with the time of acetylation being held constant at 45 minutes. The temperatures used in the acetylations were 58° F., 64° F., 70° F., 76° F., and 82° F.

All acetylations and presoakings were carried out in a one-pound package dyeing machine modified in such a manner that the above temperatures were easily obtainable and held constant.

The acetylated cotton was analyzed for acetyl content, fiber fineness, tensile strength and uniformity of acetylation. The values of the acetyl content were plotted against the times and temperatures of presoaking and the temperatures of acetylation. These graphs were analyzed to determine the effect of time and temperature of presoaking on the rate and degree of acetylation and the effect of the temperature of acetylation on the degree of acetylation of the different varieties of cotton fibers.

Table 1. Summary of Physical and Chemical Properties of the Original Cotton Fibers

Cotton	% Moisture Content	Micro- naire Mg/inch	Pressley Index	Angle 40%*	Maturity(%) NaOH Method*
Memphis	5.8	2.45	7.15	37.92	38
Empire Bale 92 Bale 249290	5.8	3.70	7.30	34.08	72
Bob Shaw	5.4	5.10	8.42	31.32	88**
Stoneville 2B	5.9	3.20	8.07	31.20	80
Acala 1517	6.0	4.00	8.93	29.76	86
Lockett 140	5.9	5.67	7.51	36.18	92

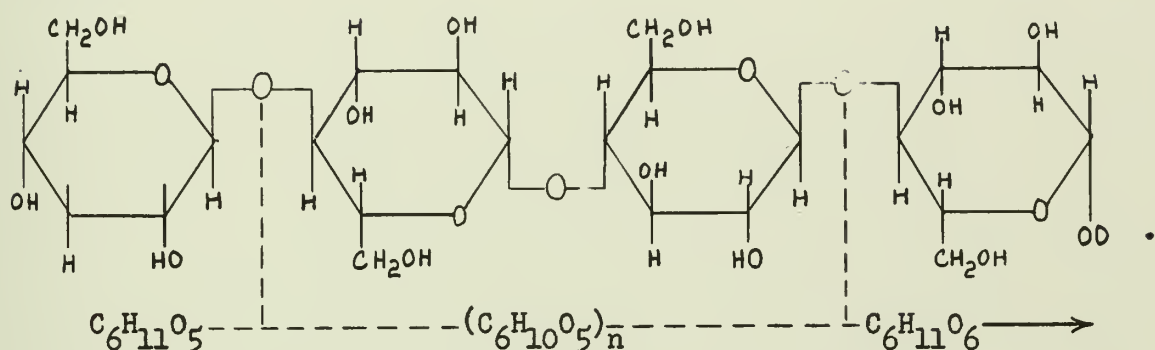
*These results were furnished by the Southern Regional Research Laboratory, New Orleans, La.

**Maturity was determined by the Areatometer.

CHAPTER II

THEORETICAL CONSIDERATIONS

The structure of the cellulose molecule may be represented as follows:¹



The glucose units, $\text{C}_6\text{H}_7\text{O}_2(\text{OH})_3$, of which the cellulose chain is composed, contains three hydroxyl groups which are free to react to form cellulose derivatives. The acetylation of cellulose is an example of esterification and consists of replacing the hydroxyl groups by acetyl groups. The number of hydroxyl groups replaced in a particular reaction will determine the degree of modification of the cellulose and the attendant changes in properties.²

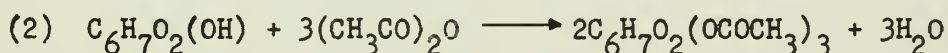
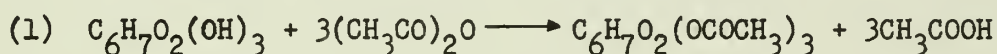
The acetylation of cotton fibers with glacial acetic acid alone occurs very slowly, even under the effects of high temperatures. Malm

¹Petrus H. Hermans, *Physics and Chemistry of Cellulose Fibres*. New York: The Elsevier Publishing Co., Inc., 1949, p. 8.

²Charles F. Goldthwait, "Acetylation to Make a New Textile," *Yearbook of Agriculture*, 1950-51, p. 421.

and Clark³ boiled surgical cotton in glacial acetic acid and at the end of 96 hours only 6.2% acetyl content was reached. Even after 500 hours only 6.4% acetyl content was obtained and that with considerable degradation to the fibers. In sealed glass tubes at 140°-145° C., 7.5% acetyl content was obtained after 48 hours. Again the long time and high temperature led to considerable degradation. The use of acetic anhydride in place of glacial acetic acid results in greatly increased acetyl content, 38.26% at 118° C., but again the cellulose is considerably degraded.⁴ Heuser and his coworkers⁵ have shown that the degradation of cellulose depends upon the time and temperature of acetylation independently of acetyl values reached.

Cellulose acetate thus cannot be prepared by direct esterification with glacial acetic acid, instead it is necessary to use acetic anhydride. The reaction may be expressed by either of the following equations:⁶



The theoretical anhydride requirement in the first case is 189 per cent, whereas in the second case it is only one-half this amount. Since a con-

³C. J. Malm and H. T. Clark, "The Action of Fatty Acids on Cellulose," Journal of the American Chemical Society, 51 (January, 1929), pp. 274-77.

⁴Emil Heuser, The Chemistry of Cellulose. New York: John Wiley and Sons, Inc., 1947, pp. 227-28.

⁵Emil Heuser and others, "Acetylation of Cellulose in Phosphoric Acid Solution," Industrial and Engineering Chemistry, 40 (1948), 1500-06.

⁶Emil Heuser, op. cit., p. 230.

siderable excess of 189 per cent is actually required for complete substitution, it is probable that the reaction is more truly expressed by the first equation although it is difficult to decide just which reaction is more likely.

The acetylation reaction is greatly facilitated by the use of certain catalysts, such as sulphuric and perchloric acids, which are actually dehydrating agents although they do not seem to act as such when acetic anhydride is used for acetylation. The function of the catalyst is not clearly understood. One theory advanced in the case of sulfuric acid is that the acid reacts with the acetic anhydride to form acetylsulfuric acid which functions as the acetylating agent and regenerates sulphuric acid to form a complete cycle. The chief function of the sulfuric acid seems to be as an aid in the swelling of the cellulose material and to degrade it.⁷

In this study perchloric acid, which is the most powerful catalyst for the acetylation of cellulose,⁸ was used. In acetic acid/acetic anhydride systems, strong acids such as perchloric acid exhibit "super-acidity" which may be accounted for by postulating the existence of highly acidic ions and in this respect perchloric acid is stronger than sulfuric acid.⁹ In acetylation, perchloric acid first forms an addition complex with the cotton; this is slowly broken down by the introduction of acetyl

⁷Emil Heuser, op. cit. pp. 228-232.

⁸Emil Heuser, op. cit. p. 240.

⁹H. A. E. Mackenzie, "Acetic Acid/Acetic Anhydride System: Properties," Transactions of the Faraday Society, 44 (1948), 159-71.

groups and at the same time small amounts of perchloric ester groups are formed. Theoretically the triacetate would thus contain no perchloric acid.¹⁰

The amount of catalyst used effects the rate of acetylation, thus with an increase in the amount of catalyst, the rate of acetylation increases.¹¹ However the amount of absorbed catalyst is not as important as the uniformity of its distribution in the cellulose. High reactivity of cellulose towards acetylation is obtained where there is a rapid and uniform absorption of the catalyst.¹²

The rate of acetylation is greatly influenced by the moisture content of the cellulosic material. The rate of reaction increases with increasing moisture content until the maximum is reached at about 25 to 30 per cent. The increasing water content causes the capillaries of the fibers to enlarge which favors the diffusion of the acetylating mixture into the inner parts of the fiber. When the moisture content reaches 25 to 30 per cent the fibers have reached saturation, i.e. all capillaries are filled with water, so that further increase of water content has no effect upon the diffusion and hence upon the rate of reaction.¹³ Any

¹⁰Genevieve Petitpas, "The Perchloric Acid Catalyst of the Acetylation of Cellulose," Mem. services chim etat., 31 (1944), 178-86, as abstracted in Chemical Abstracts, 40-2 (1946), 7613.

¹¹Emil Heuser, op. cit., p. 231.

¹²C. J. Malm and others, "Treatment of Cellulose Prior to Acetylation," Industrial and Engineering Chemistry, 44 (1952), 2904-09.

¹³Emil Heuser, op. cit., pp. 243-45.

compound which enlarges the lattice spacings and permits faster diffusion into the crystallite increases the reaction rate.¹⁴

Presoaking with glacial acetic acid has the same general effect as presoaking in water. The use of glacial acetic acid is preferred in the partial acetylation of cotton as water must be completely removed prior to acetylation. Pretreatment with glacial acetic acid or water shorten the time necessary to reach a certain acetyl content and thus the degradation of the cellulose is reduced and the molecular weights of the acetates are higher.

Presoaking in water increases the strength of the cellulose acetate, the maximum strength being attained with 20 per cent moisture content. Previous swelling in glacial acetic acid however, has no effect on the strength of the cellulose.¹⁵

The effect of time and temperature of presoaking with glacial acetic acid was determined in this study.

The acetylation of cellulose is a heterogenous reaction which is due both to fiber and micellar structure. X-ray studies have shown that in the acetylation of cellulose the reagent penetrates the fiber, attacking first the surface of the micelle which is changed into the triacetate and then proceeds inwards, producing between the unreacted interior and the completely reacted surface a partially reacted area in which only

¹⁴W. A. Sisson, "X-Ray Diffraction Behavior of Cellulose Derivatives," Industrial and Engineering Chemistry, 30 (May, 1938), p. 532.

¹⁵E. Elod, H. Schmid-Bielenberg and I. Thoria, "Acetylation of Cellulose Fibres," Angew Chemistry, 47 (1934), 465-68, as abstracted in Journal of the Textile Institute, 25 (1934), A420.

one or two hydroxyl groups have been replaced.¹⁶ In the crystalline structure where the cellulose chains are densely packed and regularly oriented the reaction is slow. In the amorphous portion of the fiber, where access of the reagents is greater due to the expanded structure, the reaction is much faster. No definite border exists between these regions but transitional regions exist in an intermediate degree.¹⁷ Thus the amorphous regions are accessible for chemical reactions as well as the surface of the crystalline regions.¹⁸ X-ray studies indicate the crystalline fraction of cotton to be 70 per cent within small experimental error.¹⁹

Sisson²⁰ states that the kinetics of cellulose derivative formation are determined largely by the velocity of diffusion rather than by the speed of reaction. The rate in the first stage is thus determined by the nature of the intermicellar spaces through which the reagent becomes accessible to the micellar surfaces. In the second and final phase, the reaction progresses from the surface of the micelle to the interior. Here the rate depends on the velocity of diffusion of the reagent through the reacted surface layer to the unattacked center of the micelle with

¹⁶W. A. Sisson, op. cit., p. 531.

¹⁷Alexander Meller, "Reactivity of Fibrous Cellulose," Journal of Polymer Science, 4 (1949), 619-28.

¹⁸L. Ward, "Crystallinity of Cellulose and Its Significance for the Fiber Properties," Textile Research Journal, 20 (1950), 363-372.

¹⁹Petrus Hermans, op. cit., p. 316.

²⁰W. A. Sisson, op. cit., p. 531.



the reaction taking place at the boundary surface between these two portions. At this stage, the rate of reaction depends primarily on the permeability of the reacted layer to the reactant molecules.

The reactions of cellulose according to Heuser²¹ may be divided into four possibilities which are derived from the physical and chemical reactivities of the hydroxyl groups. First, the reagent displays an exclusive preference for one of the two types of hydroxyl groups and the reaction proceeds in a topochemical fashion. A break in the rate of reaction occurs when all of the reactive and easily accessible hydroxyl groups have reacted and the reagent begins to penetrate the denser crystalline regions. Second, the reaction displays a greater preference for one of the two types of hydroxyl groups than the other. The reaction proceeds in a permutoid fashion, i.e. all of the preferred hydroxyl groups react faster than the less preferred regardless of where they are located. A break in the reaction occurs when the reaction of the chemically preferred hydroxyl groups approaches completion and the other reaction of the less preferred groups becomes more pronounced. Third, the reagent exerts no preference for any of the hydroxyl groups. A break in the reaction occurs when all the hydroxyl groups located in the amorphous region have reacted and the reagent begins to enter the crystalline regions. Fourth, the reagent shows no preference either chemically for any of the hydroxyl groups or physically for amorphous regions. The reagent overcomes the barriers of the microscopic and submicroscopic structures and reacts quickly with

²¹Emil Heuser, "Factors which Influence the Kinetics of Cellulose Reactions," Textile Research Journal, 20 (December, 1950), pp. 363-72.



all three hydroxyl groups. The process of acetylation seems to follow the third possibility, however the actual reaction may possibly be a combination of any two or more of the above possibilities.

Cellulose reactions do not seem to follow a single pattern, the type of reaction varies depending on its reaction partner, its concentration, the reaction medium, temperature, etc. The exact pattern that the acetylation reaction follows is difficult to determine.

THE
JOURNAL OF THE
ROYAL ANTHROPOLOGICAL INSTITUTE
OF GREAT BRITAIN AND IRELAND
VOLUME 100 PART 1
2000

CONTENTS

1

2

3

4

5

6

7

8

CHAPTER III

INSTRUMENTATION AND EQUIPMENT

Morton one-pound Package Dyeing Machine modified with cooling coils for temperature control (See Figs. I and II)

Brabender Moisture Tester

Sheffield Micronaire, Model No. 60600

Pressley Fiber Strength Tester

Forced Circulation, electrically heated Oven

Laboratory Cutting Mill, Willey Intermediate Model

Precision Balance

Kodak Timer

Glacial Acetic Acid

Acetic Anhydride

Perchloric Acid, 60%

Ammonium Hydroxide, U.S.P., 28%

Hydrochloric Acid, C.P.

Sodium Hydroxide, C.P.

Methyl Alcohol, Anhydrous, Acetone Free

Sodium Sulfate, Anhydrous

Triton X-100 (Wetting Agent)

Dyestuffs

Chlorantine Fast Blue 3RRL (Ciba)

Celliton Fast Yellow RRA (Gen. Dyestuff Corp.)

Other standard laboratory equipment and materials

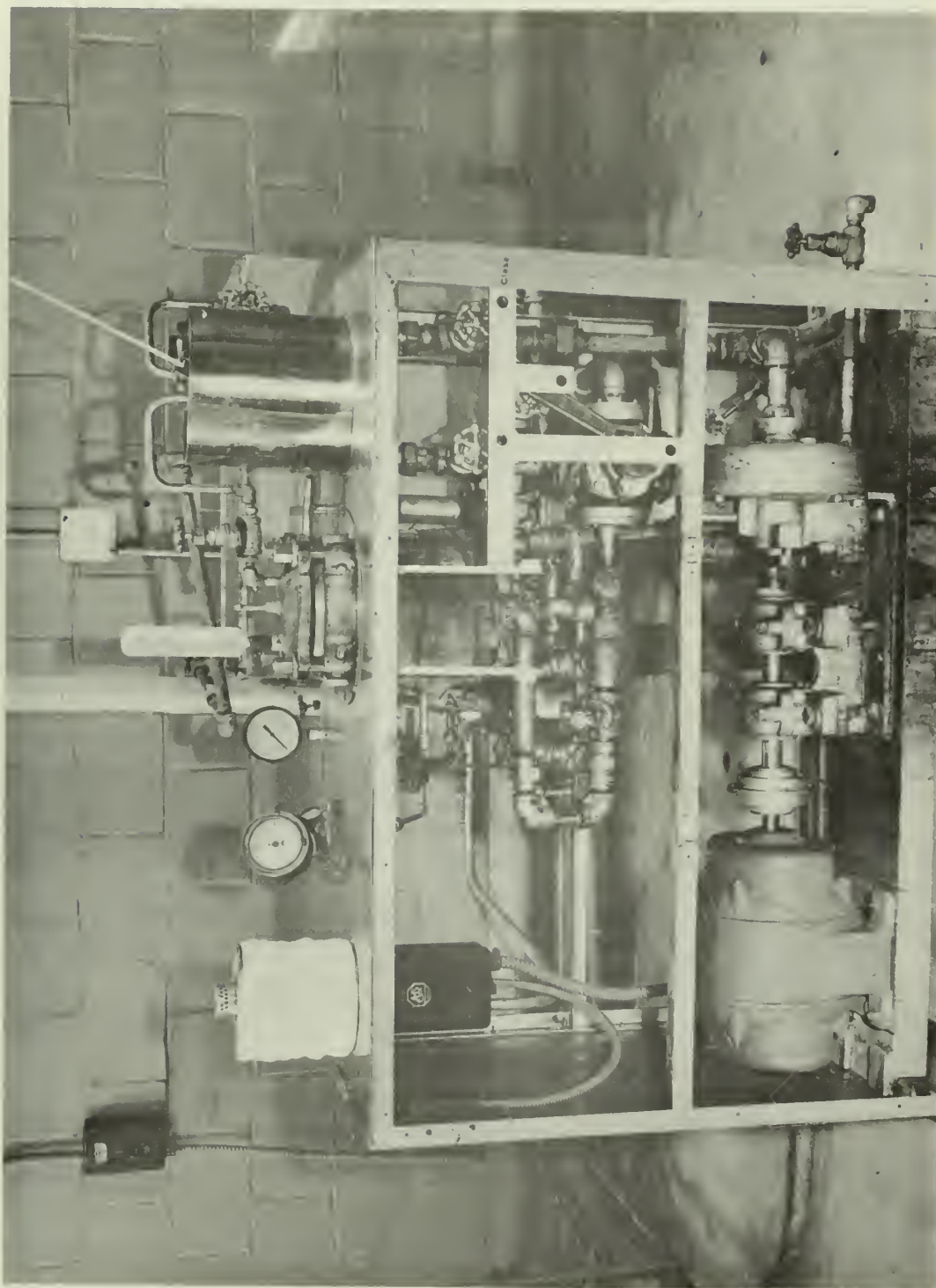


Figure 1. Front View of the Acetylation Machine.



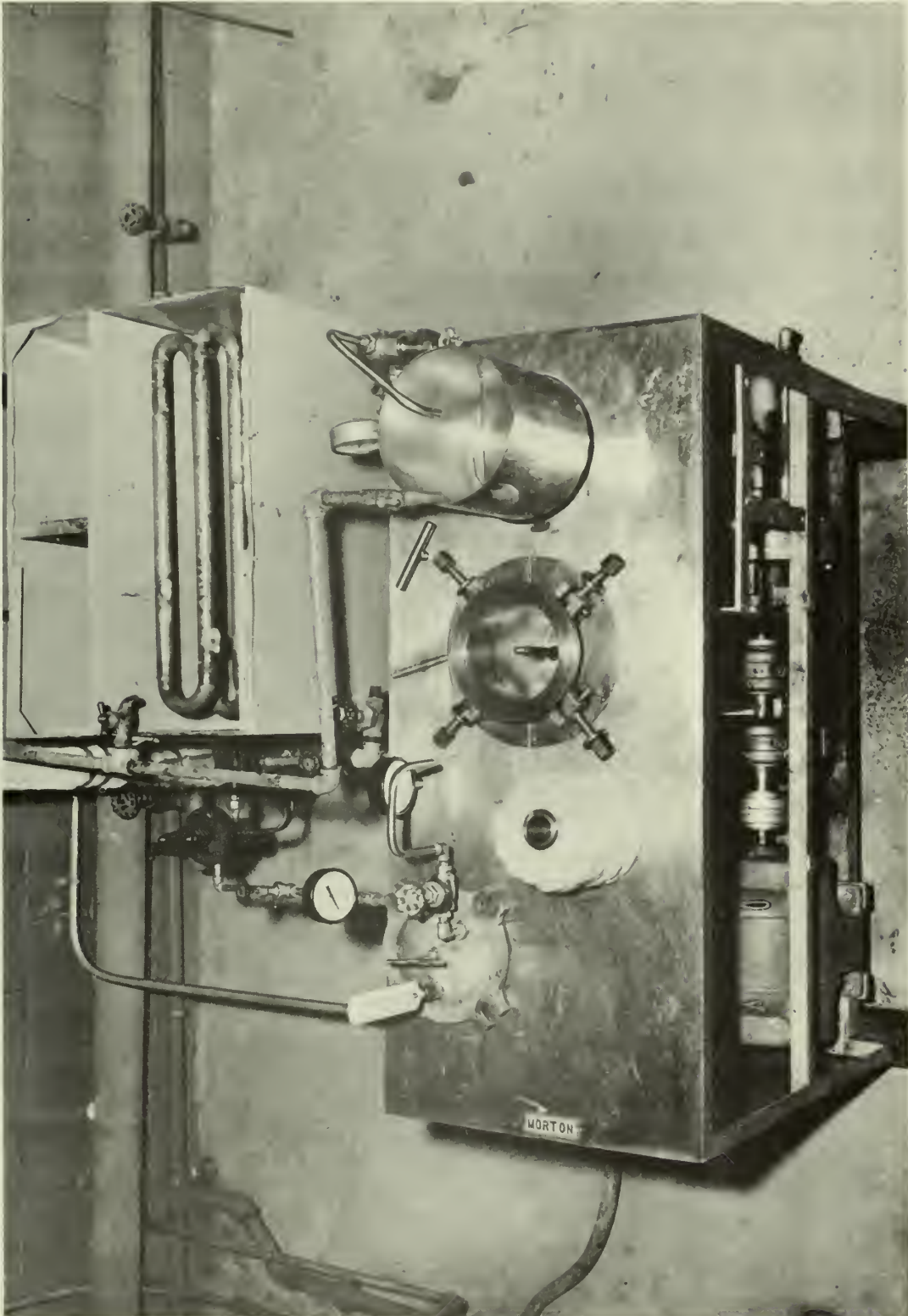


Figure 2. Top View of the Acetylation Machine.

CHAPTER IV

EXPERIMENTAL PROCEDURE

Selection of Cotton Fibers

Six varieties of cotton fibers were selected for this study. These cottons were selected on the basis of possessing a wide range of variables such as maturity, fiber fineness and tensile strength. The cottons selected were Memphis, Empire Bale, Bob Shaw 92, Stoneville 2B, Acala 1517, and Lockett 140.

Testing Prior to Acetylation

Moisture Content - Brabender.--The per cent moisture contained in the cotton was determined using the Brabender Moisture Tester. The cotton was conditioned for at least 48 hours at 70° F. and 65% relative humidity before being tested. The moisture tester was preheated for at least 15 minutes at 110° F. before being used. The fiber cans were placed in the machine prior to preheating. At the completion of the warm up period the perforated cans were removed from the machine and ten gram samples of conditioned cotton carefully weighed in the cans using the balance provided with the tester. The cans were then placed back into the oven of the tester and readings taken until equilibrium was established. The per cent moisture is read directly from the calibrated scale on the machine.

Fiber Fineness - Micronaire.--The fiber fineness was determined using the Sheffield Micronaire, Model No. 60600. The instrument was operated in accordance with the instruction pamphlet published by the manufacture.¹

¹Instructions For the Use of the Sheffield Micronaire, Dayton, Ohio: The Sheffield Corporation.

Fiber Strength - Pressley.--The cottons were tested for fiber strength in accordance with the method recommended by the A.S.T.M.²

Preparation of the Cotton Prior to Acetylation

Conditioning.---Prior to acetylation the cotton fibers were conditioned for at least 48 hours at 70° F. and 65 per cent relative humidity. After conditioning the required length of time the cotton was carefully weighed and placed in large manila envelopes until required for acetylation.

Presoaking.---A one-pound package dyeing machine modified with cooling coils was used for presoaking the cotton prior to acetylation. This modification consisted of adding cooling coils in series with the pipes carrying the liquid to the reaction chamber. Valves were placed in the pipe lines so that the cooling coils could be cut off from the main lines if desired. The cooling coils were placed in a large reservoir or tank in which ice and/or water could be placed. The machine was thoroughly rinsed out with used acetylation mixture prior to adding the conditioned cotton in order to be certain that the machine was free of water. The cotton to be acetylated was placed on a stainless steel spindle and stainless steel screens were placed between each variety of cotton. Six 25 gram samples plus 90 grams of filler cotton or twelve 20 gram samples were placed in the machine at a time. The total weight of cotton used was kept constant at 240 grams. Sixteen liters of glacial acetic acid was added after the cotton was placed in the machine. The

²A.S.T.M. "Tentative General Methods of Testing Cotton Fibers (No. D4114-47T)," Standards on Textile Materials, (October, 1948), p. 229.

valves were so adjusted that the cooling coils were filled with acid and could be used if so desired. The acid was adjusted to flow inside-out. The reacting chamber was steam jacketed and after the machine was filled with acid the steam (40 pounds pressure) was turned on and the acid quickly brought up to the desired presoaking temperature. The thermometer on the acetylating chamber cover was used in determining the presoaking temperature. When the desired time of presoaking was reached, the presoaking acid was quickly cooled to 80° F. through the use of the cooling coils. The cooling coils were packed with ice and water just prior to the end of the presoaking period. When the temperature of the presoaking solution reached 80° F., it was quickly drained from the machine into a five gallon acid carboy and set aside to be used for the next acetylation mixture.

Acetylation

Preparation of the Acetylation Mixture.--The acetylation mixture was composed of three parts of glacial acetic acid, one part of acetic anhydride and 0.15% of perchloric acid based on the total volume of acid and anhydride. The total volume of acetylation mixture used in the machine consisted of 12.6 liters of glacial acetic acid, 4.2 liters of acetic anhydride and 25.5 cc. of perchloric acid. The glacial acetic acid previously used for presoaking was used for acetylation. At least 12 hours prior to acetylation the acetic anhydride was added to 11.6 liters of acetic acid and the mixture allowed to stand in order that any water in the acid would be converted into acetic acid by reaction with the anhydride. The final acetylation mixture was made up just prior to the acetylation.

The acid-anhydride solution was cooled to 50° F. before adding the catalyst. The perchloric acid was mixed with the remaining liter of acetic acid and slowly added, with agitation, to the cooled acid-anhydride solution. There was an initial temperature rise of about 4°-8° F. when the perchloric acid was added after which mixture was again cooled to 50° F.

Acetylation.--Upon completion of presoaking and after draining the machine of the acid, the acetylating mixture was added to the machine through the reserve tank. The valve to the cooling coils was opened and the acetylating solution was pumped through the coils. The valves to the cooling coils were manipulated in such a manner that the temperature was held constant during the acetylation. All acetylations were carried out for 45 minutes; at the end of which time the acetylation mixture was quickly drained and the cotton thoroughly washed. In part I of this study the temperature of acetylation was held constant at 64° F. and in part II the temperature was varied in six degree increments from 58° F. to 82° F. for the different acetylations.

Treatment After Acetylation

Washing.--After the machine was drained the samples were washed with cold water. The washing was continued for 30 minutes after which the machine was drained. The machine was again filled with water and 50 cc. of concentrated ammonium hydroxide added and circulated through the cotton for about ten minutes or until tests with litmus paper indicated the wash water was basic. The machine was again drained and cold water

circulated with the drain valve opened until tests with litmus paper indicated the water passing out of the machine was neutral. The machine was then drained and the cotton removed.

Drying.--The excess water was removed from the acetylated cotton by the use of a hydro-extractor. The samples were then dried in hot circulating air at 180° F.

Conditioning.--Upon completion of drying the acetylated cotton was conditioned at 70° F., and 65 per cent relative humidity for at least 48 hours prior to making any analysis.

Testing After Acetylation

Physical Properties.--The moisture content, fiber fineness and fiber strength of the acetylated cottons were determined using the procedures previously described.

Analysis for Acetyl Content.--Two to three grams of the conditioned samples of acetylated cotton were carefully handpicked from the original 20 or 25 gram samples and ground up in the Wiley Mill. They were then transferred into tared 250 ml. Erlenmeyer flasks. The flasks were again accurately weighed and the exact weight of the samples calculated. From the per cent moisture previously determined, the dry weight of the samples were calculated. Fifty milliliters of 75% methyl alcohol were added to each sample and heated for 15-20 minutes at 140° F. Fifty milliliters of approximately 0.5 N sodium hydroxide solution was then added to each flask and the flasks again heated to 140° F. and allowed to stand 12 to 24 hours before

titrating. Two blanks of unacetylated cotton were included with each set of samples and carried through the same procedure. Two to three drops of phenolphthalein were added to the flasks as an indicator and the saponified samples then titrated with approximately 0.5 N hydrochloric acid until the pink color disappeared. If the pink color reappeared on standing more acid was used until the solution was completely neutralized. The percentage of acetyl content was then calculated according to the following equation:

Per cent acetyl content =

$$\frac{(\text{cc. acid used by blank}) - (\text{cc. acid used by sample})}{\text{Dry weight of the sample}} \times 4.302 \times N$$

where 4.302 is the milliequivalent weight of the acetyl group times 100 and N is the normality of the hydrochloric acid.

Dye Test for Evenness of Acetylation.---The uniformity of acetylation is determined qualitatively by a dyeing test. The dried samples (or portions thereof) were introduced in a bath of 70 to 1 liquor ratio containing 4% Chlorantine Fast Blue 3RRL, 4% Celleton Fast Yellow RRA and 5% Triton X-100 calculated on the weight of the sample. After dyeing for 30 minutes at 180°-190° F., 50% of anhydrous sodium sulphate (on the weight of the samples) was added and the dyeing continued for 30 minutes more. The samples were then removed from the dye bath and washed well with cold water and then dried in the oven at 210°-212° F. Unacetylated cotton was dyed blue while acetylated cotton was dyed yellow. From the results of this test the evenness of the acetylation could be readily observed.

CHAPTER V

DISCUSSION OF RESULTS

In Part I of this study, the effect of time and temperature of presoaking on the rate and degree of acetylation was determined. The acetyl contents obtained at different times and temperatures are shown in Tables 4 through 7 and Figures 3 through 6.

A study of this data indicated that the temperature of the presoaking acid had a very marked effect of the degree of acetylation. Thus it can be seen that as the temperature was increased, particularly at the shorter presoaking periods, the degree of acetylation rapidly increased. This was especially true in the case of Acala 1517 Cotton as shown in Table 2 and also illustrated in figures 8 through 12 as well as the other more mature fibers.

Table 2. A Comparison of the Acetyl Content of
Acala 1517 Cotton at Various Times and Temperatures
of Presoaking

Temperature °F.	10 min. % Acetyl	30 min. % Acetyl	60 min. % Acetyl
70	2.16	2.78	4.20
100	6.61	6.78	9.75
130	9.80	12.73	15.05
170	14.01	14.45	14.42

The first part of the paper discusses the importance of the study and the objectives of the research. It also outlines the methodology used in the study and the results obtained. The second part of the paper discusses the implications of the study and the conclusions drawn from the research.

The study was conducted in a laboratory setting and the results were compared with those obtained from previous studies. The study found that the results were consistent with those obtained from previous studies and that the methodology used in the study was effective.

The study also found that the results were consistent with those obtained from previous studies and that the methodology used in the study was effective. The study found that the results were consistent with those obtained from previous studies and that the methodology used in the study was effective.

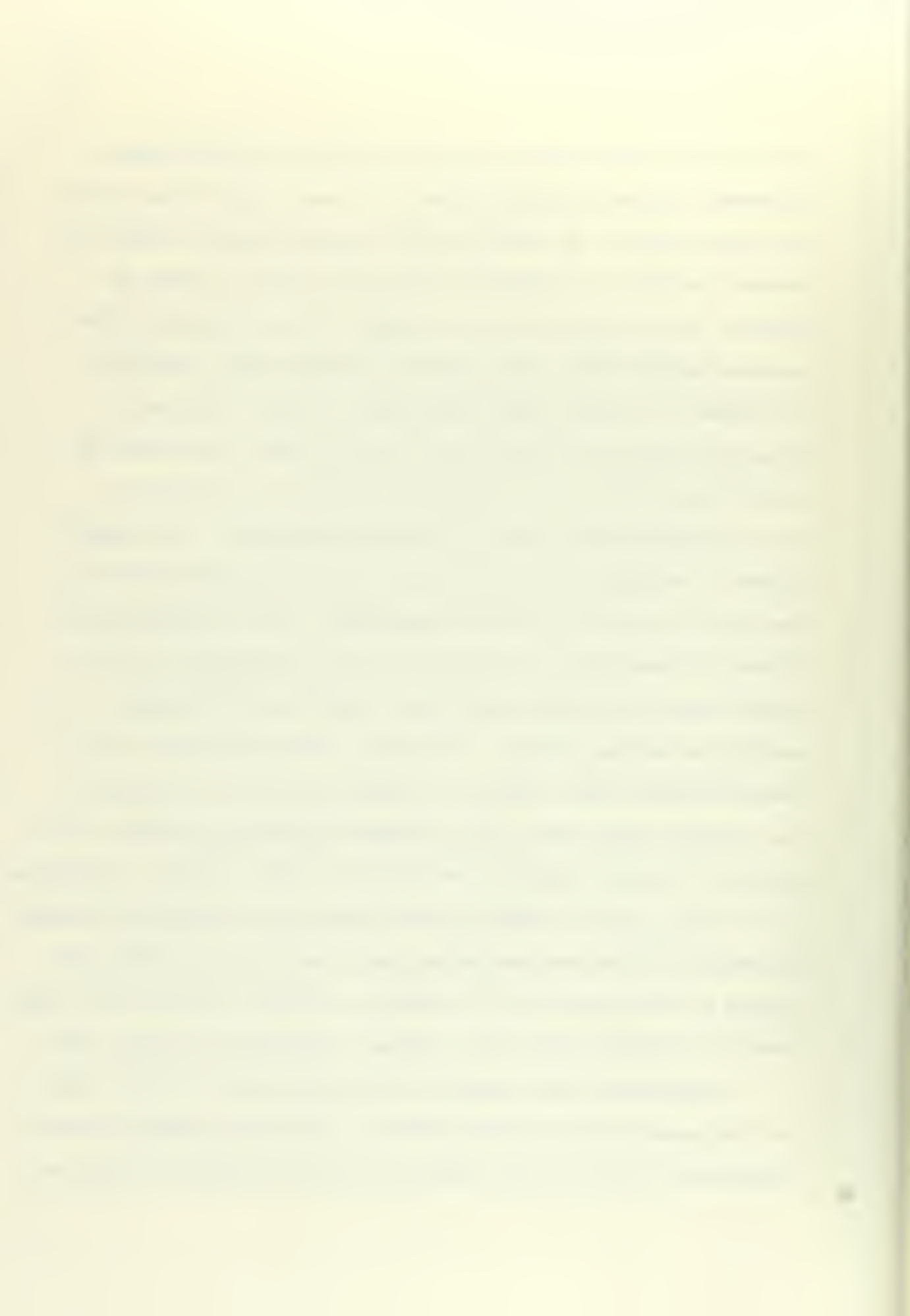
The study found that the results were consistent with those obtained from previous studies and that the methodology used in the study was effective. The study found that the results were consistent with those obtained from previous studies and that the methodology used in the study was effective.

The study found that the results were consistent with those obtained from previous studies and that the methodology used in the study was effective. The study found that the results were consistent with those obtained from previous studies and that the methodology used in the study was effective.

The study found that the results were consistent with those obtained from previous studies and that the methodology used in the study was effective. The study found that the results were consistent with those obtained from previous studies and that the methodology used in the study was effective.

As the time of presoaking increased, the effect of the temperature decreased, with the exception of the 70° F. run. This is shown by the fact that at 100° F. the acetyl content reached a maximum in about 120 minutes, at 130° F. in about 60 minutes and at 170° F. in about 30 minutes. This was true for all the cottons with the exception of the immature Memphis Cotton which reached its maximum acetyl content in 10 minutes at the three higher temperatures as shown in Figure 13. After the maximum was attained there were only slight fluctuations in acetyl content with time of presoaking, which may be attributed to small variations in the cotton, experimental error, etc. The maximum degree of acetylation for each variety of fiber was approximately the same for all three of the higher temperatures. This is illustrated in Figures 13 through 18 by noting that the lines representing the three higher presoak temperatures practically coincide once the maximum acetylation has been reached. For example, the acetyl content of the immature Memphis Cotton reaches its maximum acetylation in 10 minutes and then only varies from a low of 16.07% acetyl at the 10 minute 170° F. presoak to a high of 17.99% at the 240 minute 130° F. presoak, a difference of less than 2 per cent while the most mature cotton, Lockett 140, reached its maximum at 60 minutes and varies from a low of 9.04 per cent acetyl content at the 60 minute 100° F. presoak to a high of 10.06 per cent acetyl content at the 240 minute 100° F. presoak, a difference of one per cent.

The maximum acetyl content was never established at 70° F. within the time range studied as shown in Table 3. The acetyl content was still increasing at the end of 240 minutes for all the varieties of cotton and



the degree of acetylation for the various times was considerably below that of the other temperatures. With the exception of the most immature fibers, Memphis and Empire Bale 92, the acetyl content at the end of 240 minutes was only about half the maximum obtained. The same varieties of conditioned cotton were presoaked for 18 hours at 70° F. by Blandin¹ and a comparison of his results with the acetyl values obtained for 240 minutes at 70° F. is shown below.

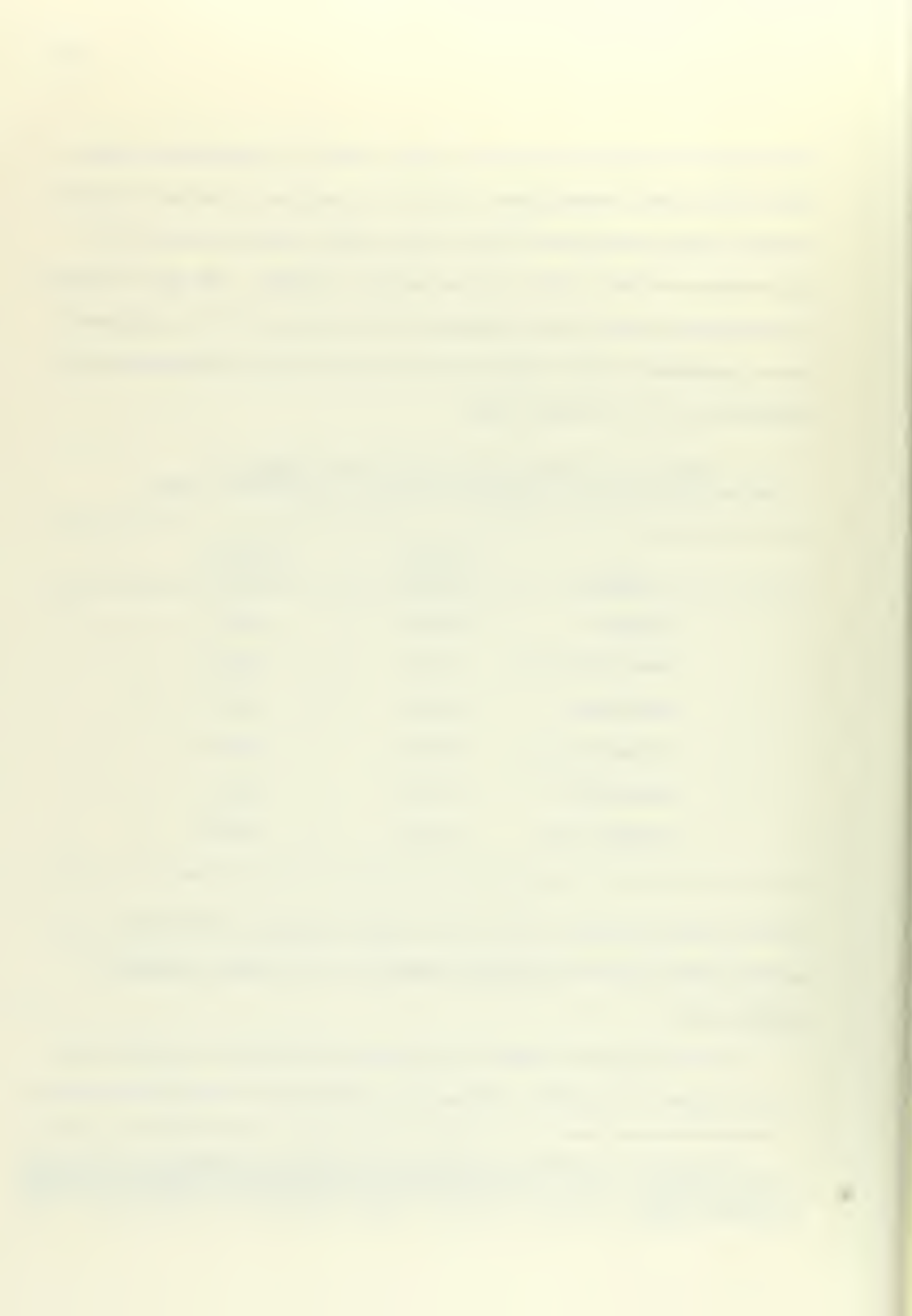
Table 3. A Comparison of the Acetyl Content of Various Cotton Fibers Presoaked at 70° F. for Different Times

Cotton	240 min. 70° F.	18 hours 70° F.
Memphis	14.44	16.75
Empire Bale 92	7.27	10.75
Bob Shaw	4.42	8.27
Stoneville 2B	8.03	12.23
Acala 1517	6.93	10.97
Lockett 140	4.00	8.56

At the end of 18 hours at 70° F. the acetyl content of the fibers were still slightly below the maximum obtained at the higher presoaking temperatures.

The hot glacial acetic acid apparently enlarges the capillaries of the fibers at a greater rate and the saturation is reached much sooner

¹Sherman W. Blandin, A Study of the Effect of Scouring and Moisture Preconditioning on the Rate and Degree of Acetylation of Various Varieties of Cotton Fibers. Master's Thesis, Georgia Institute of Technology, 1953.



thus permitting a more rapid diffusion of the acetylating mixture into the inner parts of the fiber. The higher the temperature the faster the saturation point is reached.

The different varieties of cotton reacted at different rates to the effect of the temperature of presoaking. The most immature fiber, Memphis, reached its maximum acetylation in ten minutes at the three higher presoaking temperatures while the more mature fibers, Bob Shaw and Lockett 140, took a considerably longer time, i.e. 30 minutes at 170° F., 60 minutes at 130° F., and 120 minutes at 100° F., to reach maximum acetylation. Generally speaking the more mature the fiber, the less the degree of acetylation under constant conditions. This may be shown by comparison of the acetyl content in Tables 4 through 7 with the maturity values shown in Table I. The order of increasing maturity is (1) Memphis, (2) Empire Bale 92, (3) Stoneville 2B, (4) Acala 1517, (5) Bob Shaw and (6) Lockett 140, while the order of decreasing acetyl content is (1) Memphis, (2) Acala 1517, (3) Stoneville 2B, (4) Empire Bale 92, (5) Bob Shaw, and (6) Lockett 140. Thus with the exception of Empire Bale 92 and Acala 1517 whose order is reversed, the more mature the fiber the less the acetyl content.

The higher degree of acetylation of the immature fibers is due in part to the greater percentage of amorphous structure and less per cent of crystallinity. The immature fibers exhibit greater swelling due to the collapse of the thin fiber walls and thus allowing greater diffusion of the acetylating mixture. Other factors such as gum and wax content, mineral content, alpha cellulose, etc. may also effect the degree of acetylation.

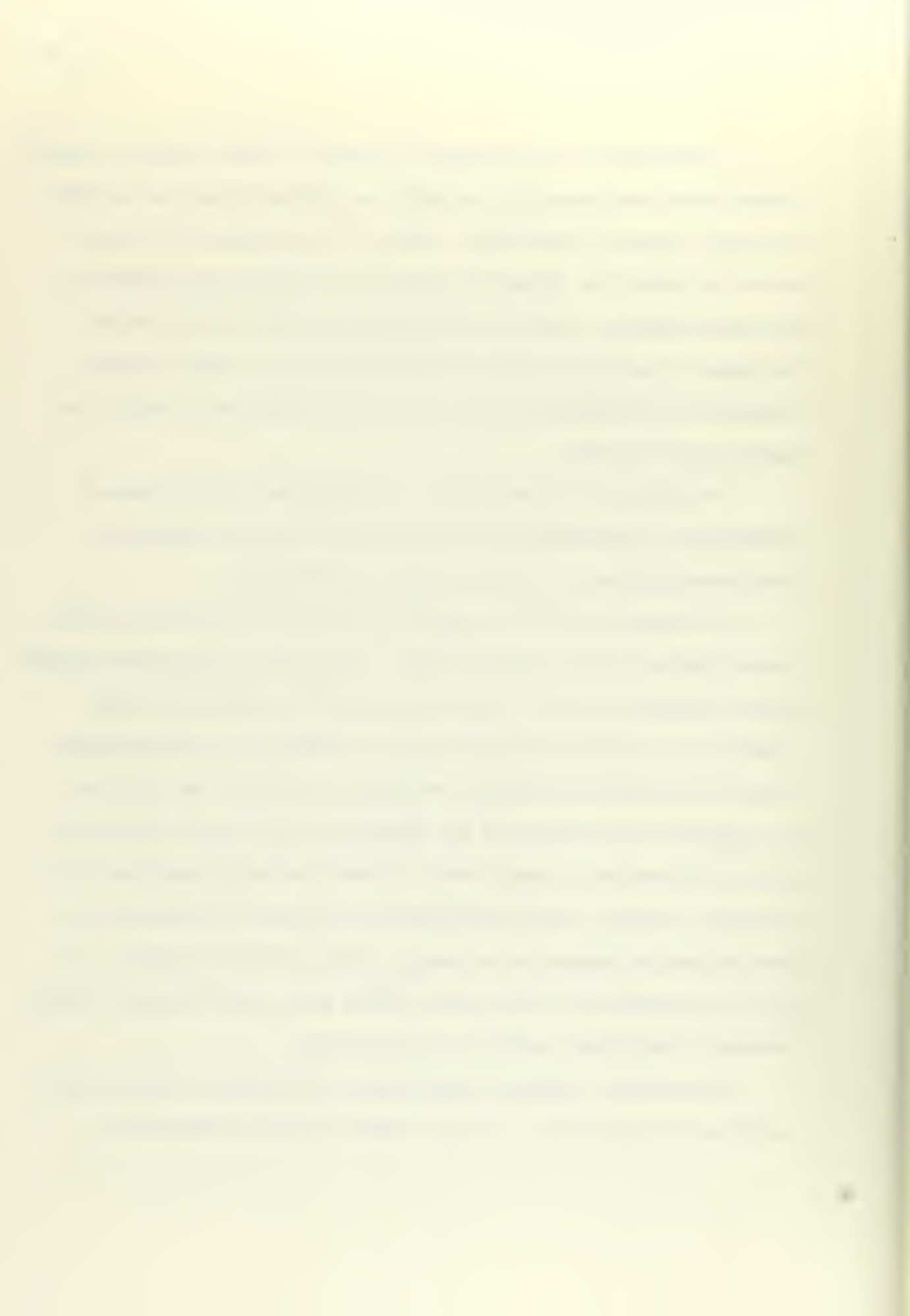


A comparison of the Micronaire Readings in Table I with the acetyl content shows that generally the higher the Micronaire Reading the lower the acetyl content of the cotton. This is to be expected as the more mature the cotton the thicker the fiber walls and thus the higher the Micronaire Reading. Both the fiber fineness and the maturity effect the degree of acetylation and both factors have to be taken into consideration in determining which cotton would be expected to exhibit the highest acetyl content.

The effect of the temperature of acetylation on the degree of acetylation is determined in Part II of this study. The results of these determinations are shown in Table 8 and Figure 7.

An increase of 24° F., from 58° F. to 82° F., doubled the acetyl values obtained for the various fibers. For example, the immature Memphis Cotton increased in acetyl content from 12.69% to 23.68%, the mature Lockett 140 cotton increased from 7.96% to 19.88%, and an intermediate cotton, Stoneville 2B increased from 10.68% to 21.51%. The effect of the temperature of acetylation was determined in six degree increments and the differences in acetyl content between increments was found to be relatively constant. The fiber degradation increased noticeably with each increase in temperature as shown in Table 23 in the appendix. At 82° F. the degradation to the cotton fibers was so great that the tensile strength of the fibers could not be determined.

The moisture content, fiber fineness, and tensile strength of the acetylated cotton samples, Tables 9 through 23, were determined by



Holloway.² It was found that as the acetyl values increased the moisture content decreased and the fiber fineness decreased (Micronaire Readings increased). The fiber strength generally decreased with increasing acetyl content and increasing time of presoaking. The tensile strength per unit weight of cellulose however, probably experiences little change except possibly in the 76° F. and 82° F. acetylations as the fiber weight increases approximately 25% at the monoacetate stage.

The dye test for evenness of acetylation produced uniform coloring throughout the cotton samples and thus showed that the acetylation was even throughout the cotton fibers.

²Benjamin Holloway, A Comparison of the Physical Characteristics of Acetylated Cotton with those of Untreated Cottons. Master's Thesis, Georgia Institute of Technology, June 1953.



Table 4. Effect of Time of Presoaking at 70° F.
on the Degree of Acetylation*

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	3.72	8.06	10.50	13.05	13.74	14.44
Empire Bale 92	1.52	2.75	3.83	5.07	6.25	7.27
Bob Shaw	0.86	1.46	2.35	3.07	4.38	4.42
Stoneville 2B	1.37	3.36	3.97	4.87	6.80	8.03
Acala 1517	1.43	2.16	2.78	4.20	5.61	6.93
Lockett 140	1.02	1.33	1.84	2.48	2.68	4.00

Table 5. Effect of Time of Presoaking at 100° F.
on the Degree of Acetylation

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	3.72	16.07	16.21	17.20	17.33	17.52
Empire Bale 92	1.52	7.87	9.10	9.86	11.77	12.27
Bob Shaw	0.86	4.38	4.92	6.44	8.45	9.34
Stoneville 2B	1.37	7.90	8.40	11.45	13.78	14.00
Acala 1517	1.43	6.61	6.78	9.75	14.30	15.40
Lockett 140	1.02	4.24	5.10	9.04	9.06	10.06

*All acetylations in Tables 4 through 7 were carried out at 64° F. for 45 minutes.

Table 1. Summary of the data collected for the study.

Variable	Mean	SD	Range	Skewness	Kurtosis	Normality
Age	35.2	12.5	18-65	0.15	-0.50	Yes
Gender	Male	Female	Male	Female	Male	Female
Education	High School	College	University	Postgraduate	High School	College
Income	Low	Medium	High	Very High	Low	Medium
Marital Status	Single	Married	Divorced	Widowed	Single	Married
Occupation	Unemployed	Self-employed	Employee	Entrepreneur	Unemployed	Self-employed
Health Status	Good	Fair	Poor	Very Poor	Good	Fair
Stress Level	Low	Medium	High	Very High	Low	Medium
Life Satisfaction	Low	Medium	High	Very High	Low	Medium

Table 2. Summary of the data collected for the study.

Variable	Mean	SD	Range	Skewness	Kurtosis	Normality
Age	35.2	12.5	18-65	0.15	-0.50	Yes
Gender	Male	Female	Male	Female	Male	Female
Education	High School	College	University	Postgraduate	High School	College
Income	Low	Medium	High	Very High	Low	Medium
Marital Status	Single	Married	Divorced	Widowed	Single	Married
Occupation	Unemployed	Self-employed	Employee	Entrepreneur	Unemployed	Self-employed
Health Status	Good	Fair	Poor	Very Poor	Good	Fair
Stress Level	Low	Medium	High	Very High	Low	Medium
Life Satisfaction	Low	Medium	High	Very High	Low	Medium

Table 3. Summary of the data collected for the study.

Variable	Mean	SD	Range	Skewness	Kurtosis	Normality
Age	35.2	12.5	18-65	0.15	-0.50	Yes
Gender	Male	Female	Male	Female	Male	Female
Education	High School	College	University	Postgraduate	High School	College
Income	Low	Medium	High	Very High	Low	Medium
Marital Status	Single	Married	Divorced	Widowed	Single	Married
Occupation	Unemployed	Self-employed	Employee	Entrepreneur	Unemployed	Self-employed
Health Status	Good	Fair	Poor	Very Poor	Good	Fair
Stress Level	Low	Medium	High	Very High	Low	Medium
Life Satisfaction	Low	Medium	High	Very High	Low	Medium

Table 6. Effect of Time of Presoaking at 130° F.
on the Degree of Acetylation

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	3.72	16.11	16.53	17.17	17.00	17.99
Empire Bale 92	1.52	8.42	10.35	12.14	12.25	12.99
Bob Shaw	0.86	6.07	8.30	9.87	10.55	11.10
Stoneville 2B	1.37	11.58	12.59	13.18	13.28	14.00
Acala 1517	1.43	9.80	12.73	15.05	15.30	16.24
Lockett 140	1.02	6.20	7.41	9.91	9.82	9.96

Table 7. Effect of Time of Presoaking at 170° F.
on the Degree of Acetylation

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	3.72	16.51	16.95	16.51	16.82	16.60
Empire Bale 92	1.52	11.41	11.95	12.08	11.82	12.53
Bob Shaw	0.86	8.89	9.52	9.62	9.95	10.45
Stoneville 2B	1.37	12.79	12.81	13.25	12.71	13.88
Acala 1517	1.43	14.01	14.45	14.42	14.96	15.36
Lockett 140	1.02	8.86	9.27	9.81	9.75	9.63

Table 1. Summary of the data for the first group of subjects.						
Subject	Age	Gender	Height (cm)	Weight (kg)	Time (min)	Score
1	25	Male	175	75	10	100
2	26	Male	178	80	12	110
3	27	Male	180	85	15	120
4	28	Male	182	90	18	130
5	29	Male	185	95	20	140
6	30	Male	188	100	22	150
7	31	Male	190	105	25	160
8	32	Male	192	110	28	170
9	33	Male	195	115	30	180
10	34	Male	198	120	32	190
11	35	Male	200	125	35	200
12	36	Male	202	130	38	210
13	37	Male	205	135	40	220
14	38	Male	208	140	42	230
15	39	Male	210	145	45	240
16	40	Male	212	150	48	250
17	41	Male	215	155	50	260
18	42	Male	218	160	52	270
19	43	Male	220	165	55	280
20	44	Male	222	170	58	290
21	45	Male	225	175	60	300
22	46	Male	228	180	62	310
23	47	Male	230	185	65	320
24	48	Male	232	190	68	330
25	49	Male	235	195	70	340
26	50	Male	238	200	72	350
27	51	Male	240	205	75	360
28	52	Male	242	210	78	370
29	53	Male	245	215	80	380
30	54	Male	248	220	82	390
31	55	Male	250	225	85	400
32	56	Male	252	230	88	410
33	57	Male	255	235	90	420
34	58	Male	258	240	92	430
35	59	Male	260	245	95	440
36	60	Male	262	250	98	450
37	61	Male	265	255	100	460
38	62	Male	268	260	102	470
39	63	Male	270	265	105	480
40	64	Male	272	270	108	490
41	65	Male	275	275	110	500
42	66	Male	278	280	112	510
43	67	Male	280	285	115	520
44	68	Male	282	290	118	530
45	69	Male	285	295	120	540
46	70	Male	288	300	122	550
47	71	Male	290	305	125	560
48	72	Male	292	310	128	570
49	73	Male	295	315	130	580
50	74	Male	298	320	132	590
51	75	Male	300	325	135	600
52	76	Male	302	330	138	610
53	77	Male	305	335	140	620
54	78	Male	308	340	142	630
55	79	Male	310	345	145	640
56	80	Male	312	350	148	650
57	81	Male	315	355	150	660
58	82	Male	318	360	152	670
59	83	Male	320	365	155	680
60	84	Male	322	370	158	690
61	85	Male	325	375	160	700
62	86	Male	328	380	162	710
63	87	Male	330	385	165	720
64	88	Male	332	390	168	730
65	89	Male	335	395	170	740
66	90	Male	338	400	172	750
67	91	Male	340	405	175	760
68	92	Male	342	410	178	770
69	93	Male	345	415	180	780
70	94	Male	348	420	182	790
71	95	Male	350	425	185	800
72	96	Male	352	430	188	810
73	97	Male	355	435	190	820
74	98	Male	358	440	192	830
75	99	Male	360	445	195	840
76	100	Male	362	450	198	850
77	101	Male	365	455	200	860
78	102	Male	368	460	202	870
79	103	Male	370	465	205	880
80	104	Male	372	470	208	890
81	105	Male	375	475	210	900
82	106	Male	378	480	212	910
83	107	Male	380	485	215	920
84	108	Male	382	490	218	930
85	109	Male	385	495	220	940
86	110	Male	388	500	222	950
87	111	Male	390	505	225	960
88	112	Male	392	510	228	970
89	113	Male	395	515	230	980
90	114	Male	398	520	232	990
91	115	Male	400	525	235	1000
92	116	Male	402	530	238	1010
93	117	Male	405	535	240	1020
94	118	Male	408	540	242	1030
95	119	Male	410	545	245	1040
96	120	Male	412	550	248	1050
97	121	Male	415	555	250	1060
98	122	Male	418	560	252	1070
99	123	Male	420	565	255	1080
100	124	Male	422	570	258	1090
101	125	Male	425	575	260	1100
102	126	Male	428	580	262	1110
103	127	Male	430	585	265	1120
104	128	Male	432	590	268	1130
105	129	Male	435	595	270	1140
106	130	Male	438	600	272	1150
107	131	Male	440	605	275	1160
108	132	Male	442	610	278	1170
109	133	Male	445	615	280	1180
110	134	Male	448	620	282	1190
111	135	Male	450	625	285	1200
112	136	Male	452	630	288	1210
113	137	Male	455	635	290	1220
114	138	Male	458	640	292	1230
115	139	Male	460	645	295	1240
116	140	Male	462	650	298	1250
117	141	Male	465	655	300	1260
118	142	Male	468	660	302	1270
119	143	Male	470	665	305	1280
120	144	Male	472	670	308	1290
121	145	Male	475	675	310	1300
122	146	Male	478	680	312	1310
123	147	Male	480	685	315	1320
124	148	Male	482	690	318	1330
125	149	Male	485	695	320	1340
126	150	Male	488	700	322	1350
127	151	Male	490	705	325	1360
128	152	Male	492	710	328	1370
129	153	Male	495	715	330	1380
130	154	Male	498	720	332	1390
131	155	Male	500	725	335	1400
132	156	Male	502	730	338	1410
133	157	Male	505	735	340	1420
134	158	Male	508	740	342	1430
135	159	Male	510	745	345	1440
136	160	Male	512	750	348	1450
137	161	Male	515	755	350	1460
138	162	Male	518	760	352	1470
139	163	Male	520	765	355	1480
140	164	Male	522	770	358	1490
141	165	Male	525	775	360	1500
142	166	Male	528	780	362	1510
143	167	Male	530	785	365	1520
144	168	Male	532	790	368	1530
145	169	Male	535	795	370	1540
146	170	Male	538	800	372	1550
147	171	Male	540	805	375	1560
148	172	Male	542	810	378	1570
149	173	Male	545	815	380	1580
150	174	Male	548	820	382	1590
151	175	Male	550	825	385	1600
152	176	Male	552	830	388	1610
153	177	Male	555	835	390	1620
154	178	Male	558	840	392	1630
155	179	Male	560	845	395	1640
156	180	Male	562	850	398	1650
157	181	Male	565	855	400	1660
158	182	Male	568	860	402	1670
159	183	Male	570	865	405	1680
160	184	Male	572	870	408	1690
161	185	Male	575	875	410	1700
162	186	Male	578	880	412	1710
163	187	Male	580	885	415	1720
164	188	Male	582	890	418	1730
165	189	Male	585	895	420	1740
166	190	Male	588	900	422	1750
167	191	Male	590	905	425	1760
168	192	Male	592	910	428	1770
169	193	Male	595	915	430	1780
170	194	Male	598	920	432	1790
171	195	Male	600	925	435	1800
172	196	Male	602	930	438	1810
173	197	Male	605	935	440	1820
174	198	Male	608	940	442	1830
175	199	Male	610	945	445	1840
176	200	Male	612	950	448	1850
177	201	Male	615	955	450	1860
178	202	Male	618	960	452	1870
179	203	Male	620	965	455	1880
180	204	Male	622	970	458	1890
181	205	Male	625	975	460	1900
182	206	Male	628	980	462	1910
183	207	Male	630	985	465	1920
184	208	Male	632	990	468	1930
185	209	Male	635	995	470	1940
186	210	Male	638	1000	472	1950
187	211	Male	640	1005	475	1960
188	212	Male	642	1010	478	1970
189	213	Male	645	1015	480	1980
190	214	Male	648	1020	482	1990
191	215	Male	650	1025	485	2000
192	216	Male	652	1030	488	2010
193	217	Male	655	1035	490	2020
194	218	Male	658	1040	492	2030
195	219	Male	660	1045	495	2040
196	220	Male	662	1050	498	2050
197	221	Male	665	1055	500	2060
198	222	Male	668	1060	502	2070
199	223	Male	670	1065	505	2080
200	224	Male	672	1070	508	2090
201	225	Male	675	1075	510	2100
202	226	Male	678	1080	512	2110
203	227	Male	680	1085	515	2120
204	228	Male	682	1090	518	2130
205	229	Male	685	1095	520	2140
206						

Table 8. Effect of Temperature of Acetylation
on the Degree of Acetylation*

Cotton	58° F.	64° F.	70° F.	76° F.	82° F.
Memphis	12.69	16.51	19.22	20.71	23.68
Empire Bale 92	9.12	11.41	13.51	17.10	20.36
Bob Shaw	7.48	8.89	10.68	15.30	19.28
Stoneville 2B	10.68	12.79	15.40	18.31	21.51
Acala 1517	10.76	14.01	16.77	20.03	23.12
Lockett 140	7.96	8.86	10.98	14.72	19.88

*Presoaked for 10 minutes at 170° F. and acetylated for 45 minutes.

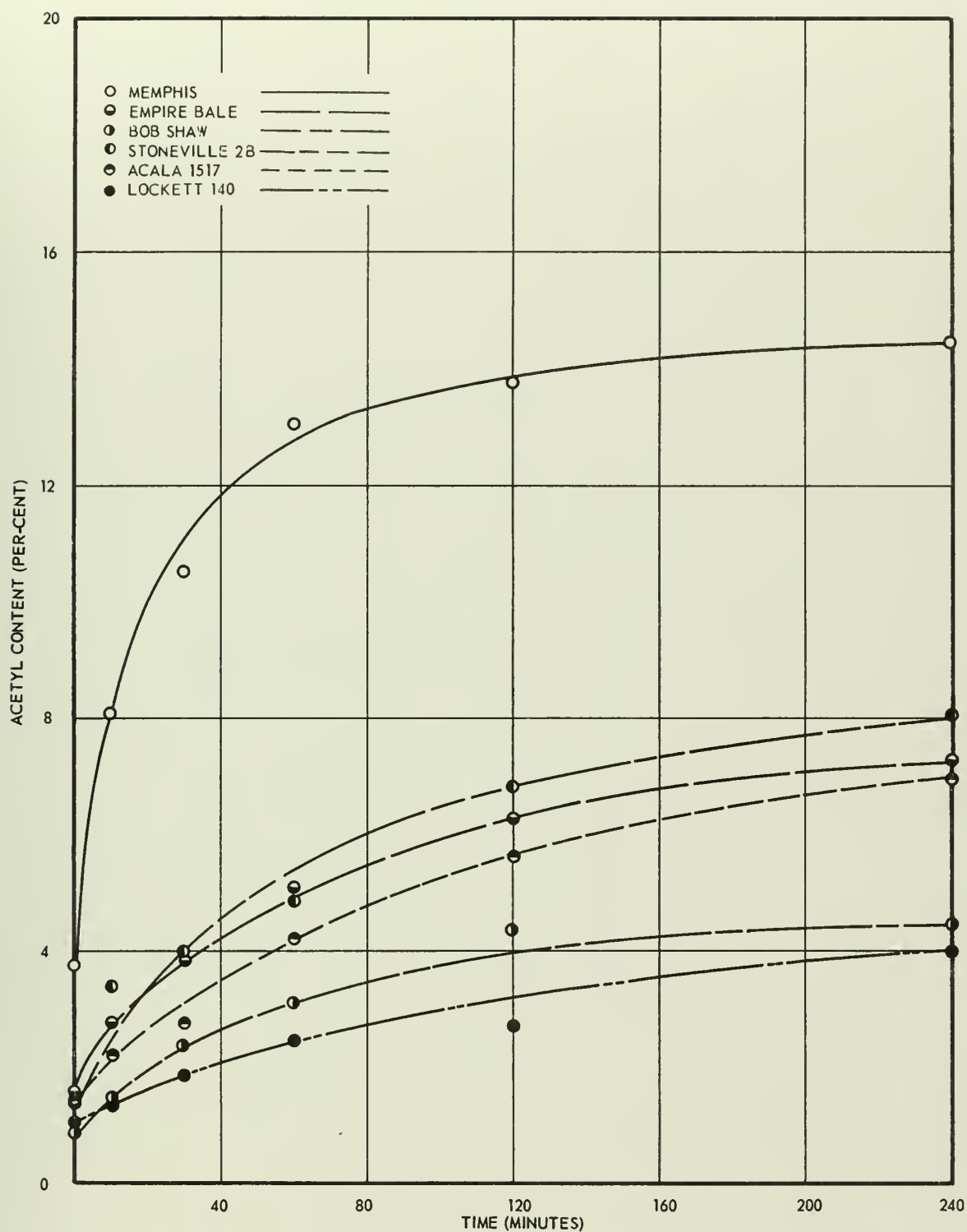


Figure 3. The Change in Acetyl Content with Presoaking Time at 70° F.



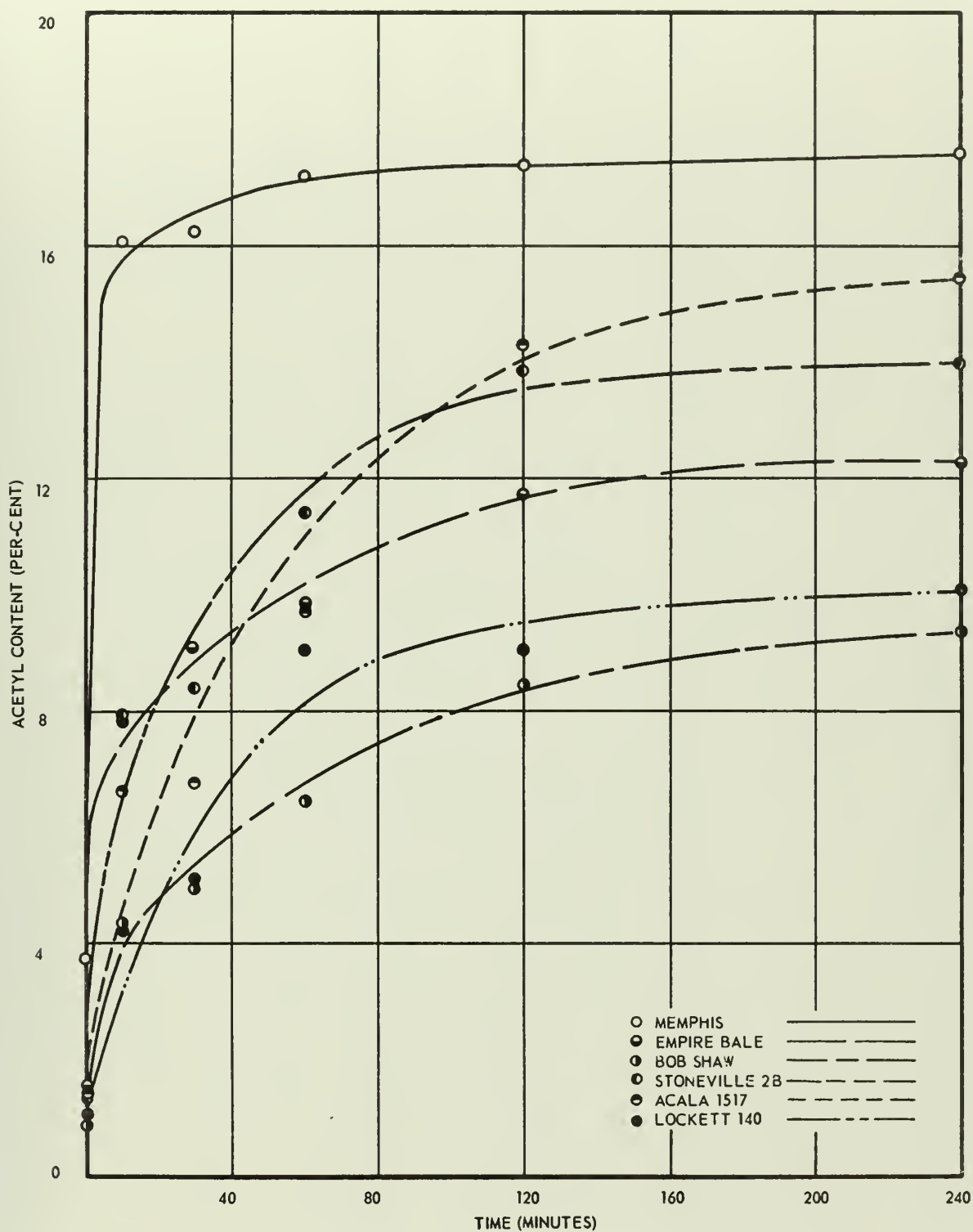


Figure 4. The Change in Acetyl Content with Presoaking Time at 100° F.



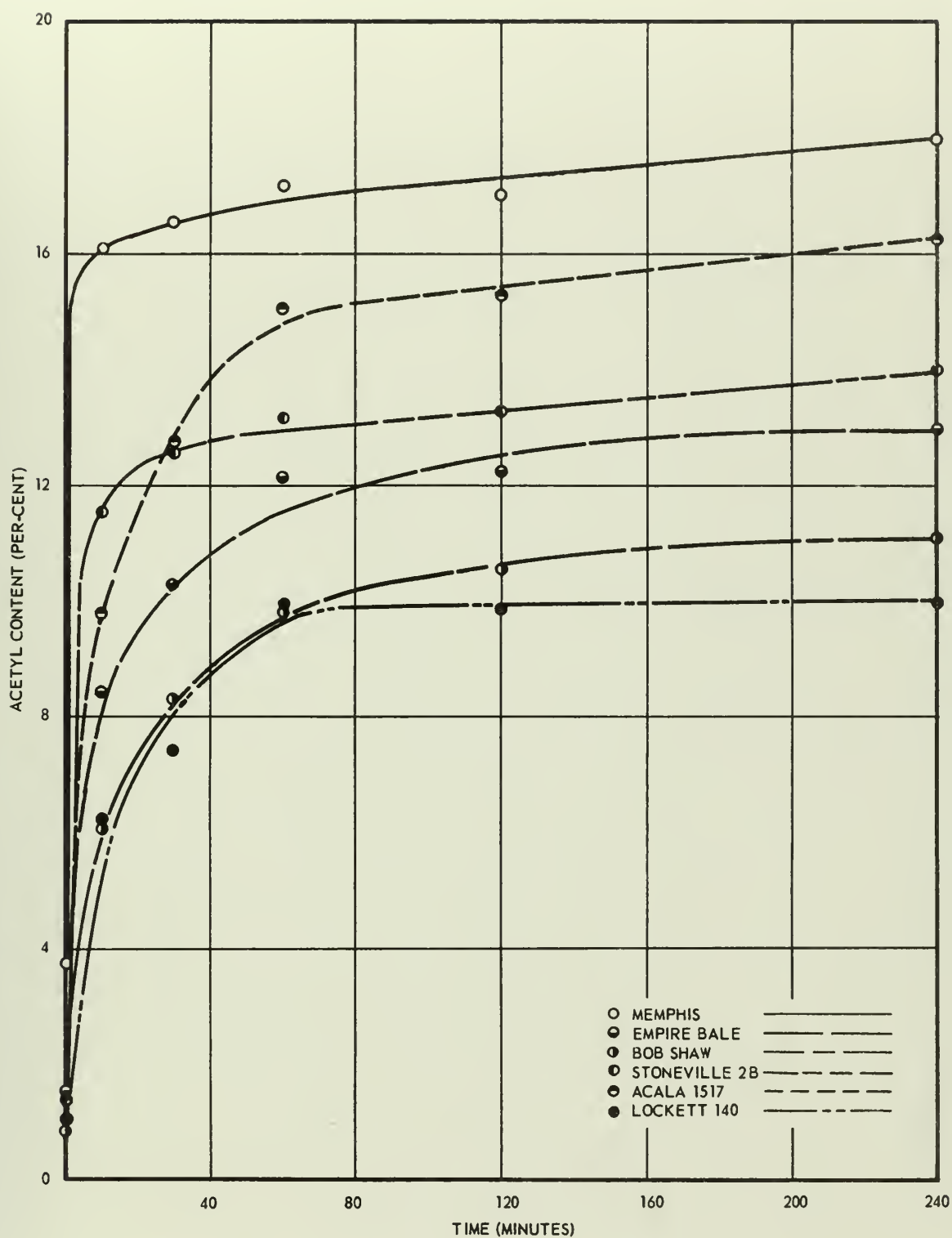


Figure 5. The Change in Acetyl Content with Presoaking Time at 130 F.



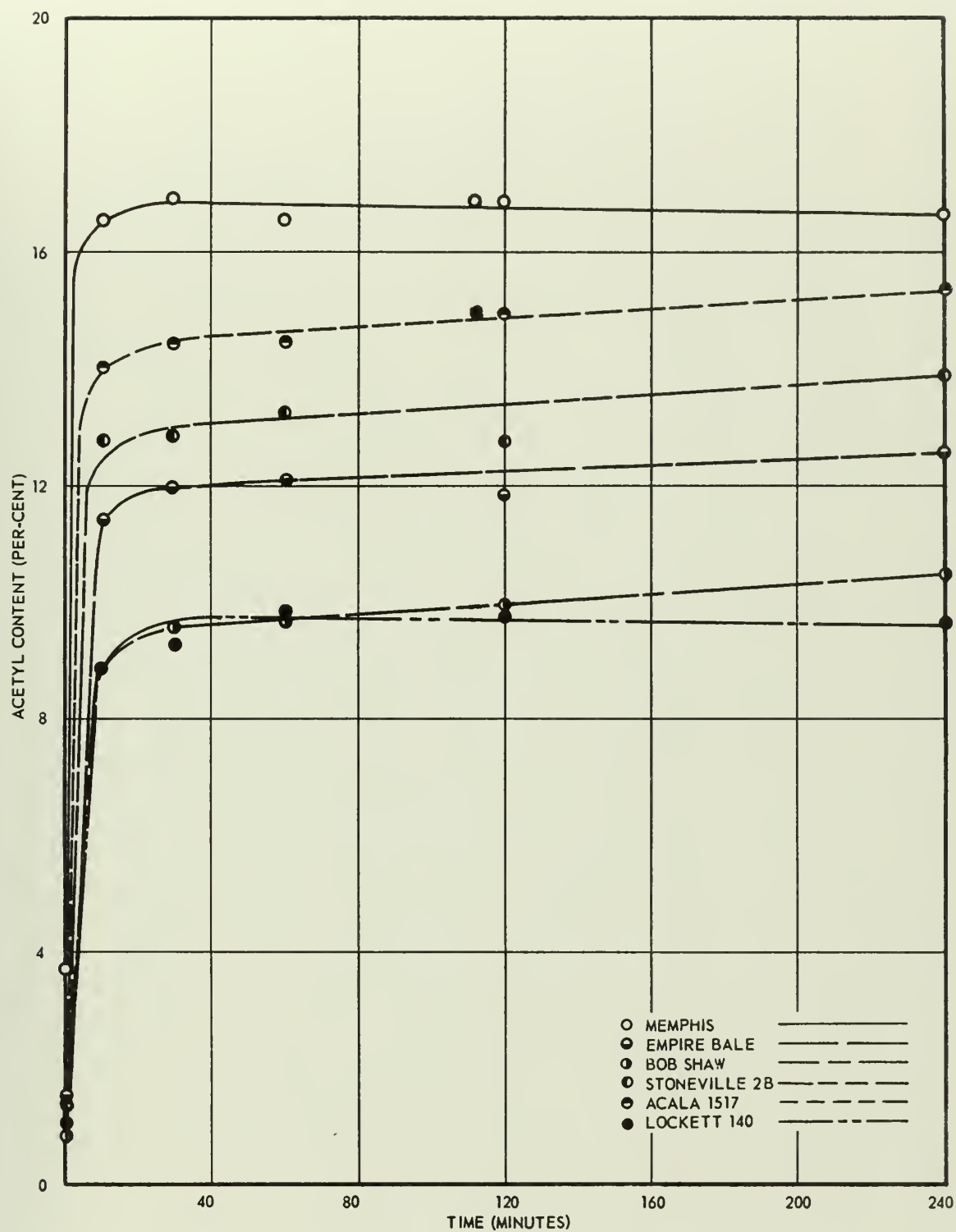


Figure 6. The Change in Acetyl Content with Presoaking Time at 170 F.

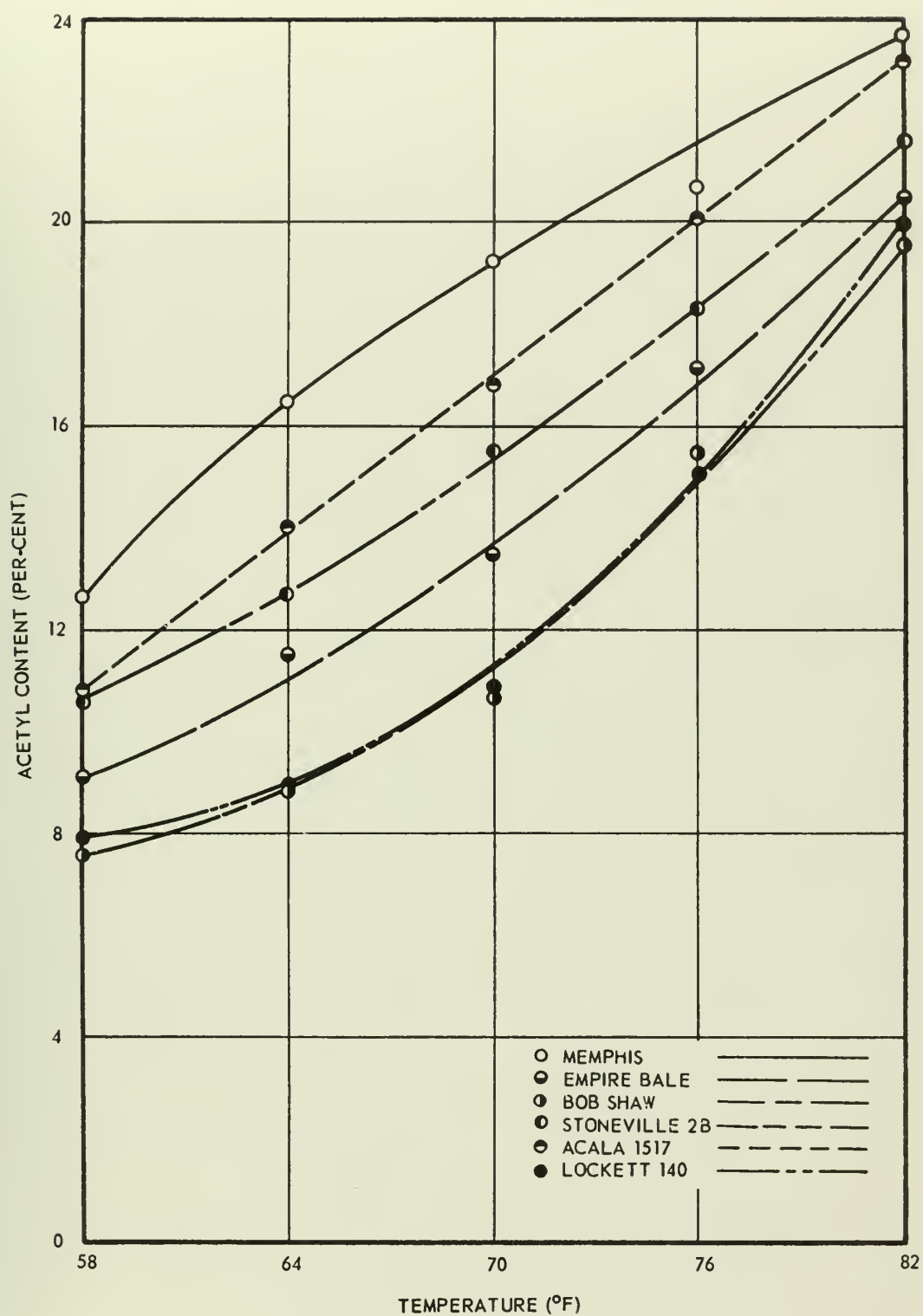


Figure 7. The Change in Acetyl Content with Acetylation Temperature using a 10 minute, 170 F. Presoak.



Figure 1

Figure 1

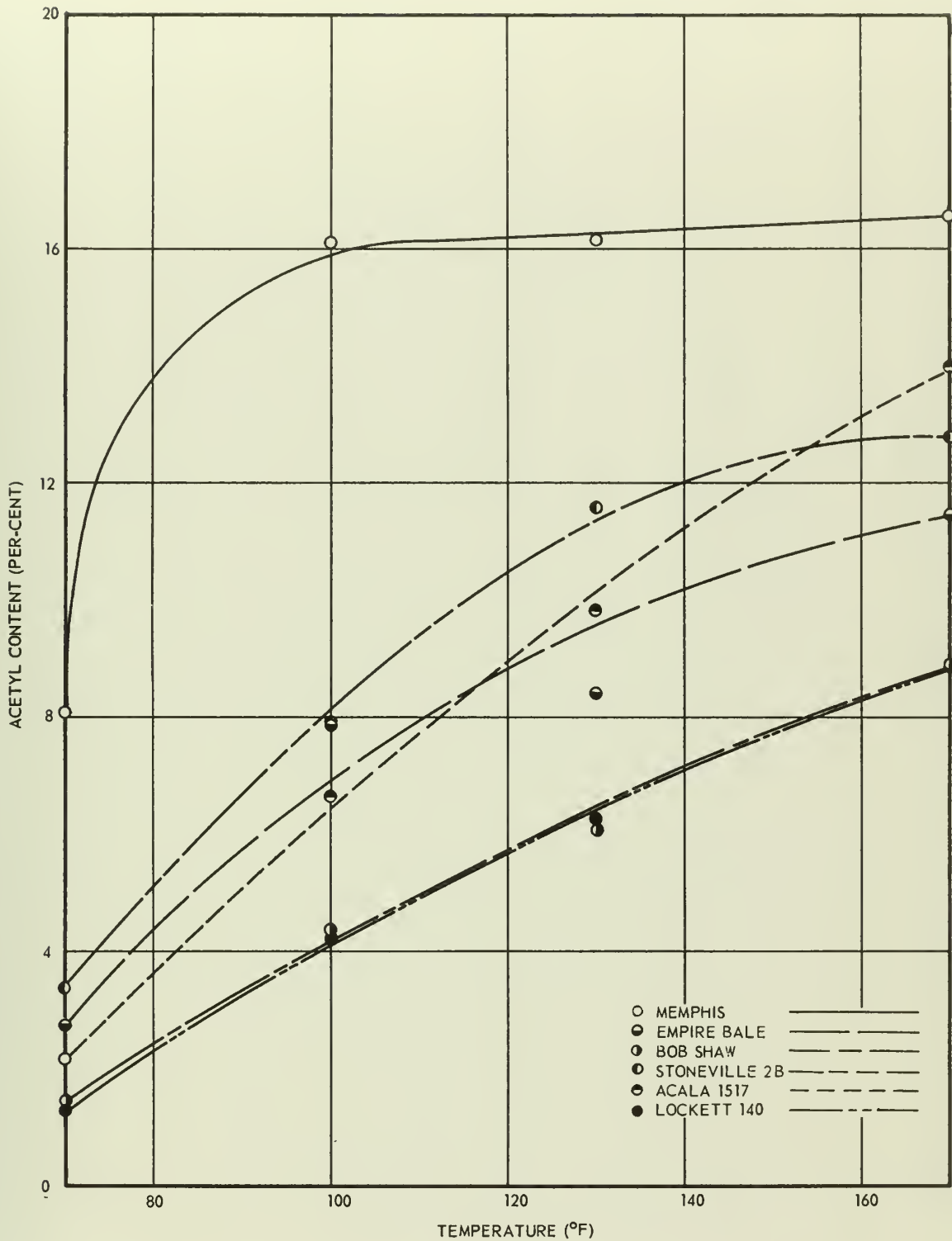


Figure 8. The Change in Acetyl Content with Presoaking Temperature using a 10 Minute Presoaking Period.



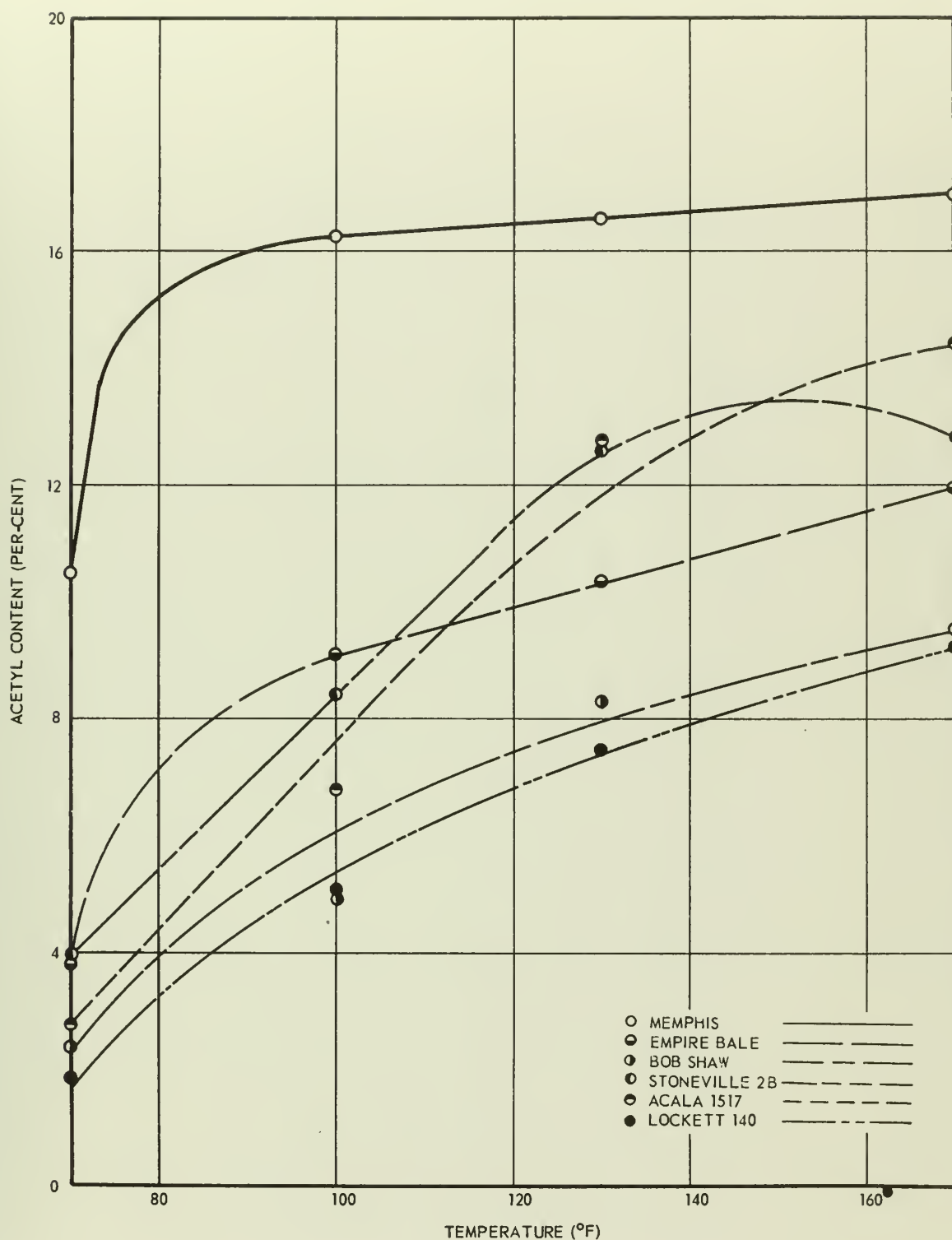


Figure 9. The Change in Acetyl Content with Presoaking Temperature using a 30 minute Presoaking Period.



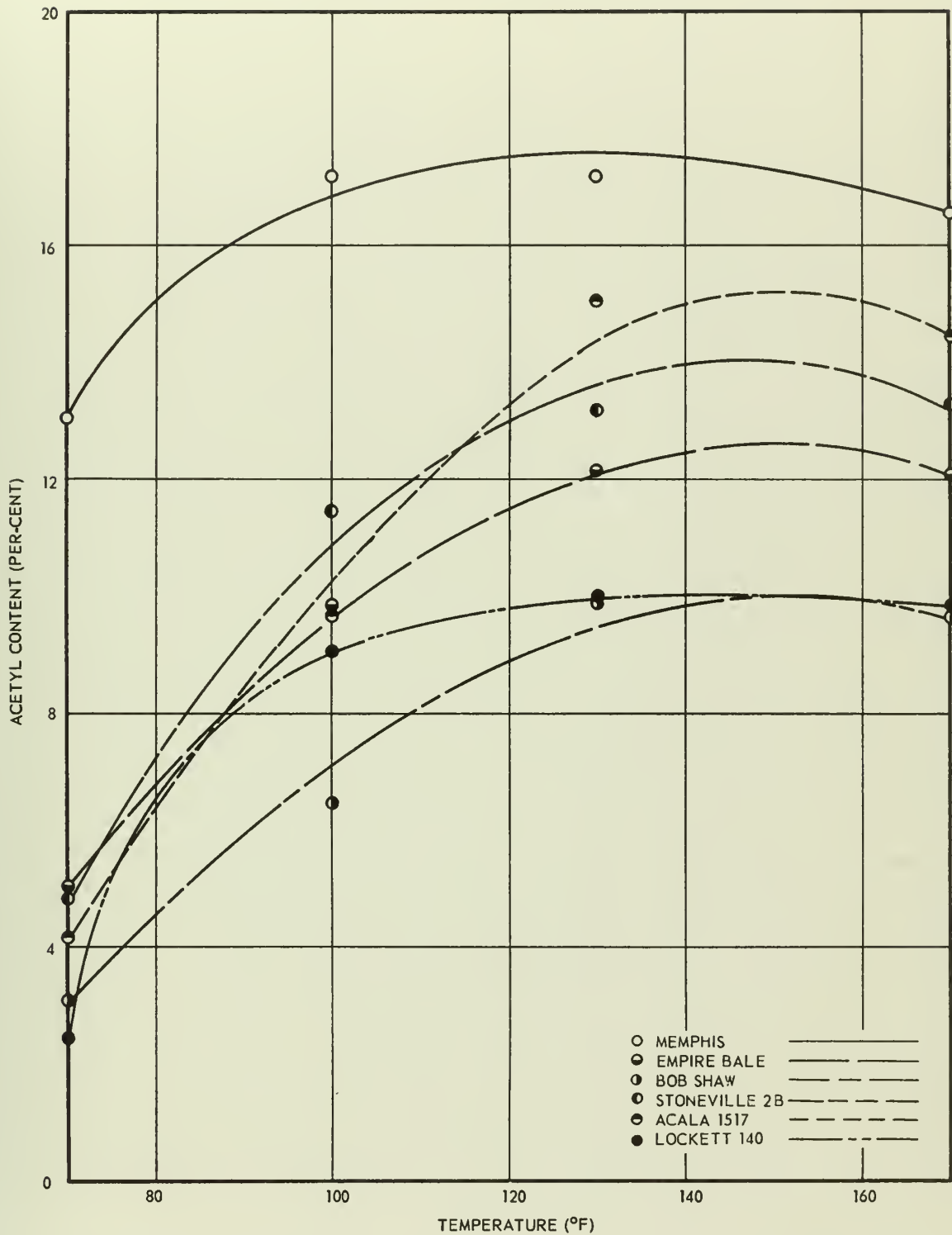


Figure 10. The Change in Acetyl Content with Fresoaking Temperature using a 60 Minute Fresoaking Period.

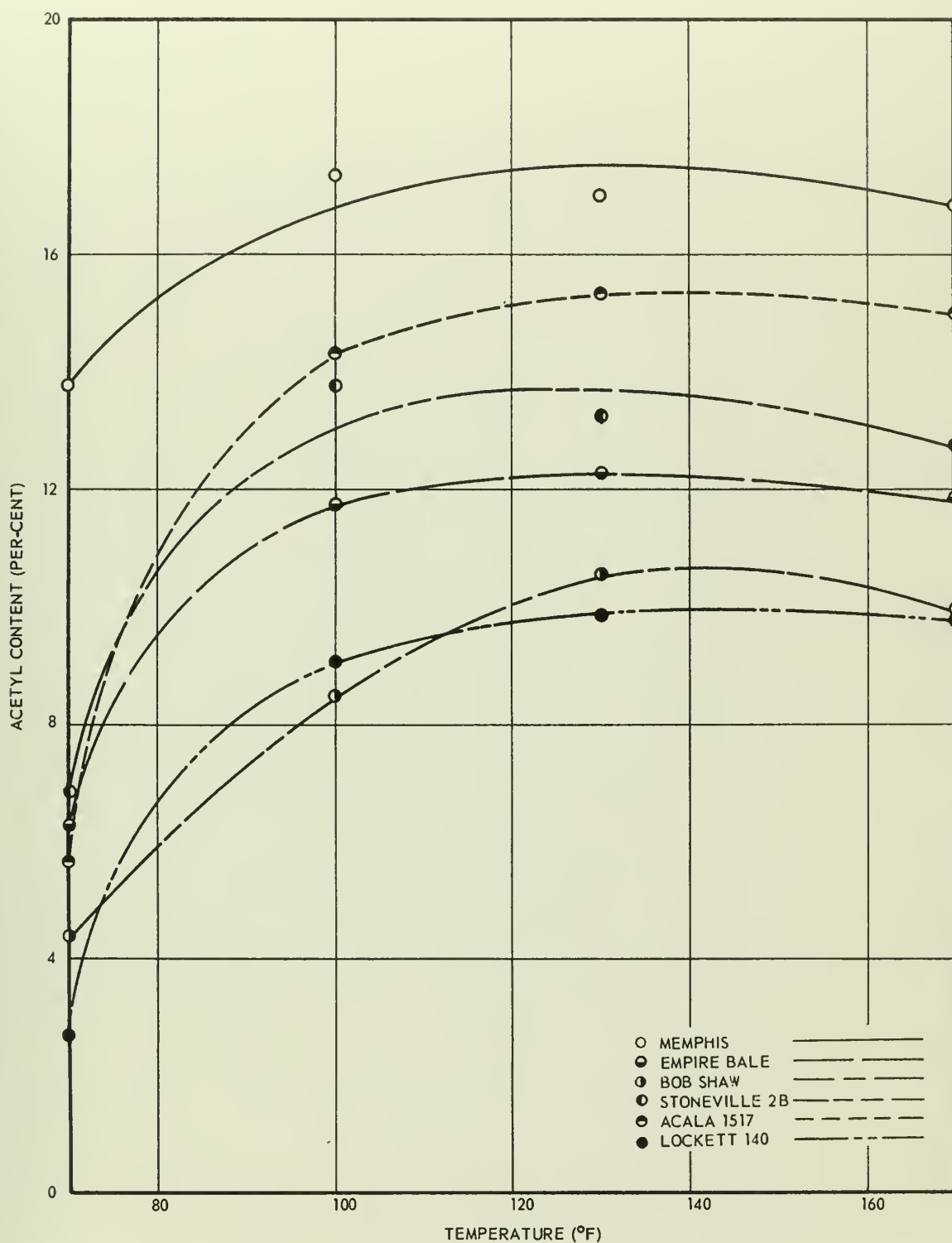


Figure 11. The Change in Acetyl Content with Presoaking Temperature using a 120 Minute Presoaking Period.



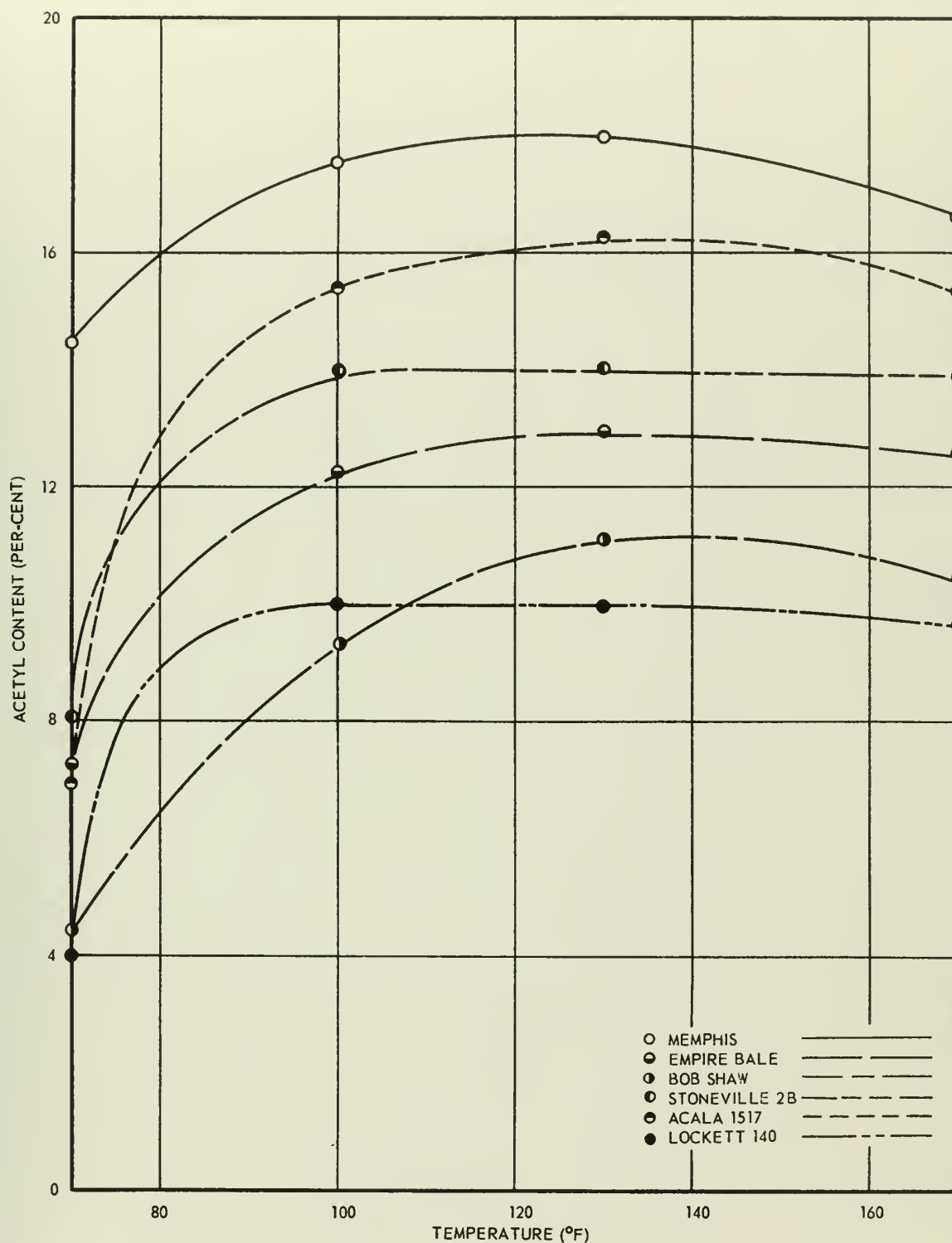


Figure 12. The Change in Acetyl Content with Presoaking Temperature using a 240 minute Presoaking Period.

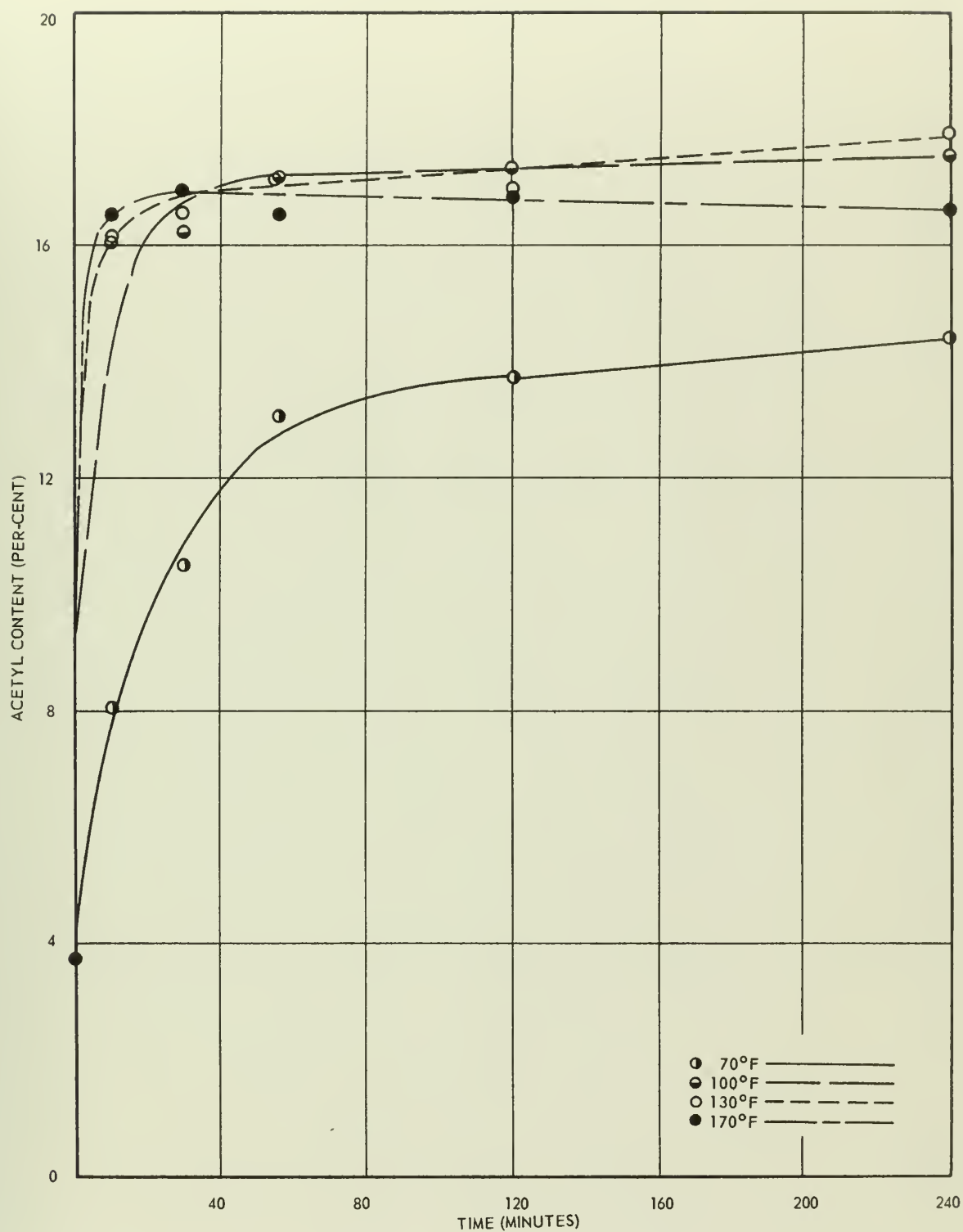


Figure 13. The Change in Acetyl Content with Presoaking Time for Memphis Cotton at Different Presoak Temperatures.



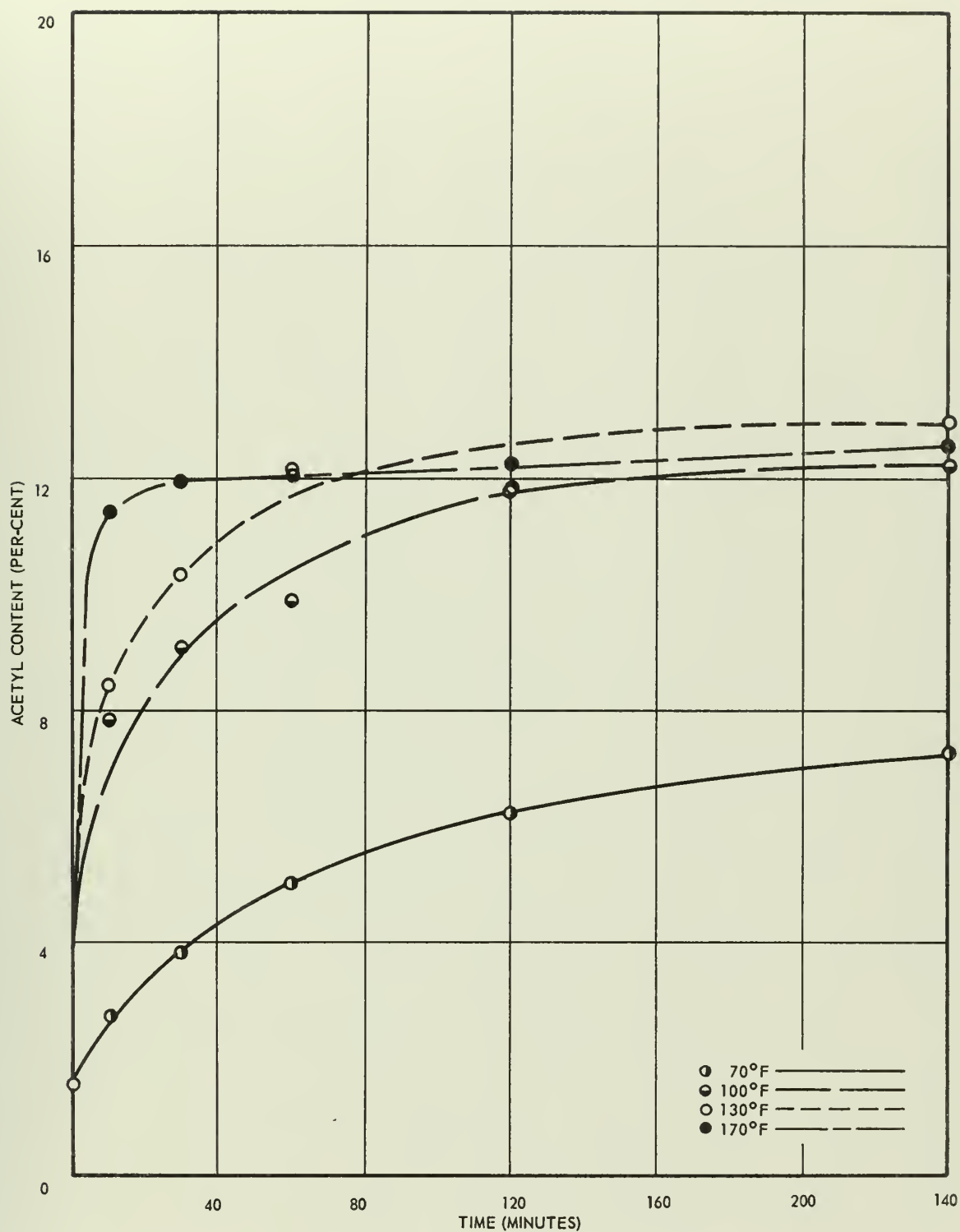


Figure 14. The Change in Acetyl Content with Presoaking Time for Empire Bale 92 Cotton at Different Presoak Temperatures.

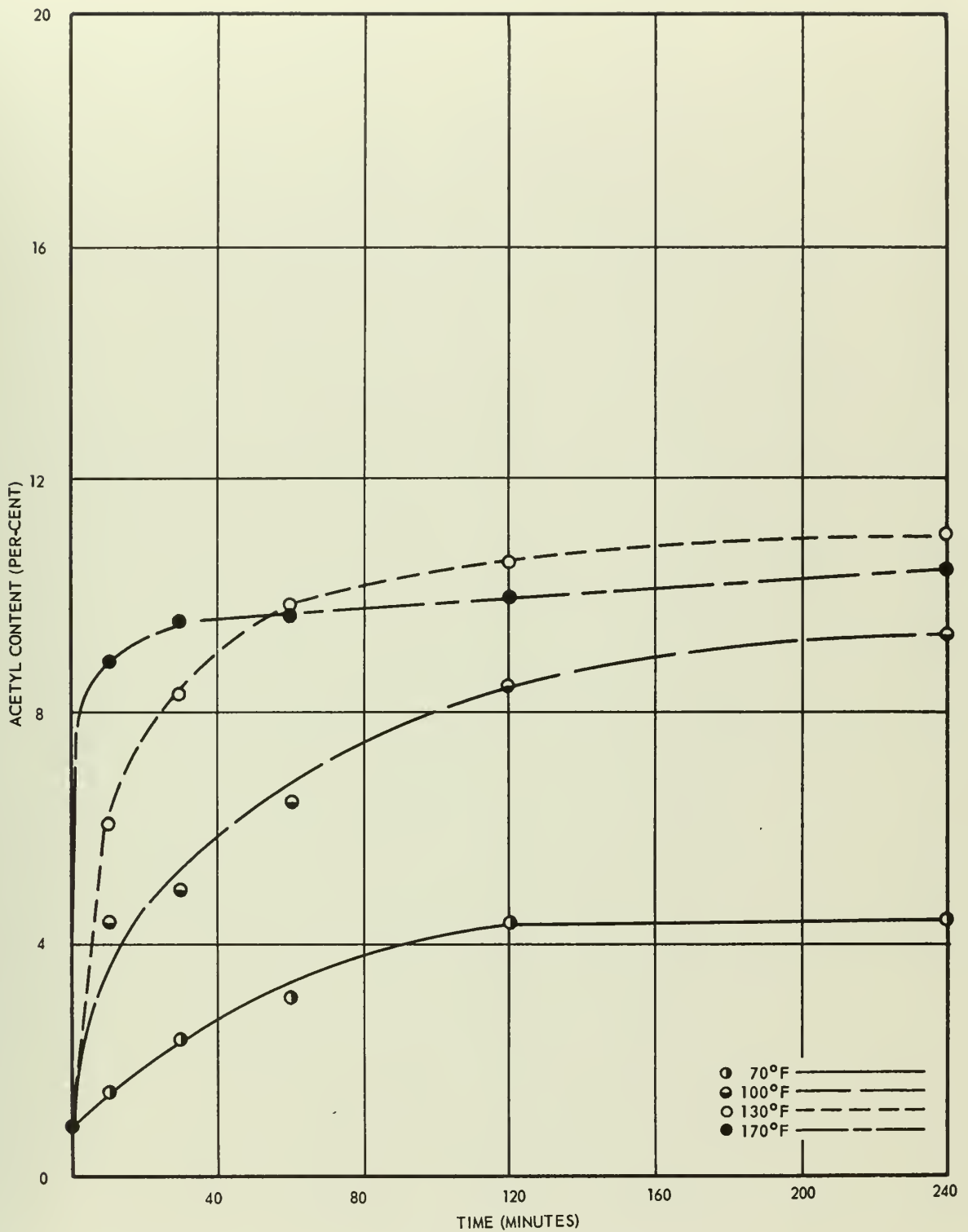


Figure 15. The Change in Acetyl Content with Presoaking Time for Bob Shaw Cotton at Different Presoak Temperatures.

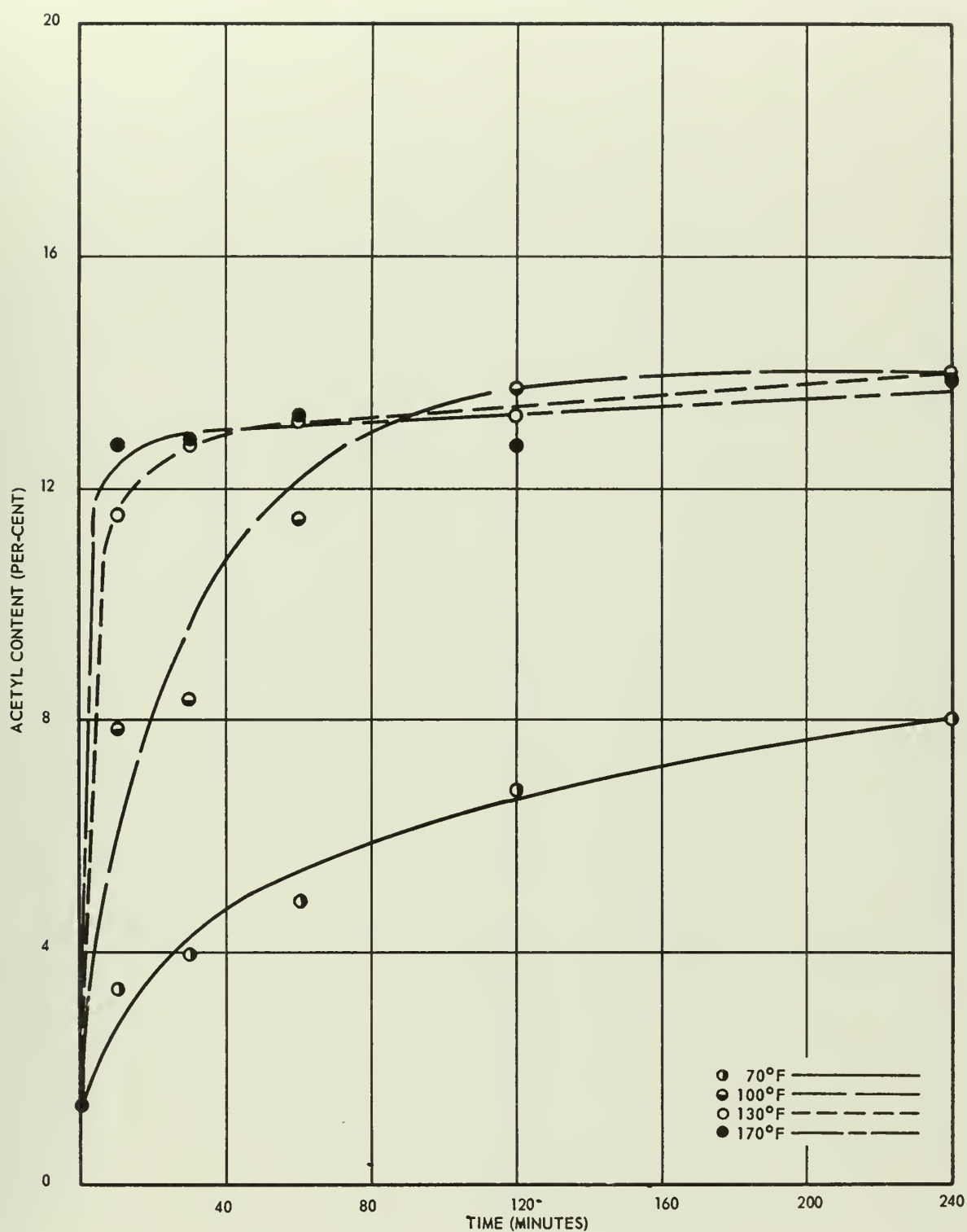


Figure 16. The Change in Acetyl Content with Presoaking Time for Stoneville 2B Cotton at Different Presoak Temperatures.

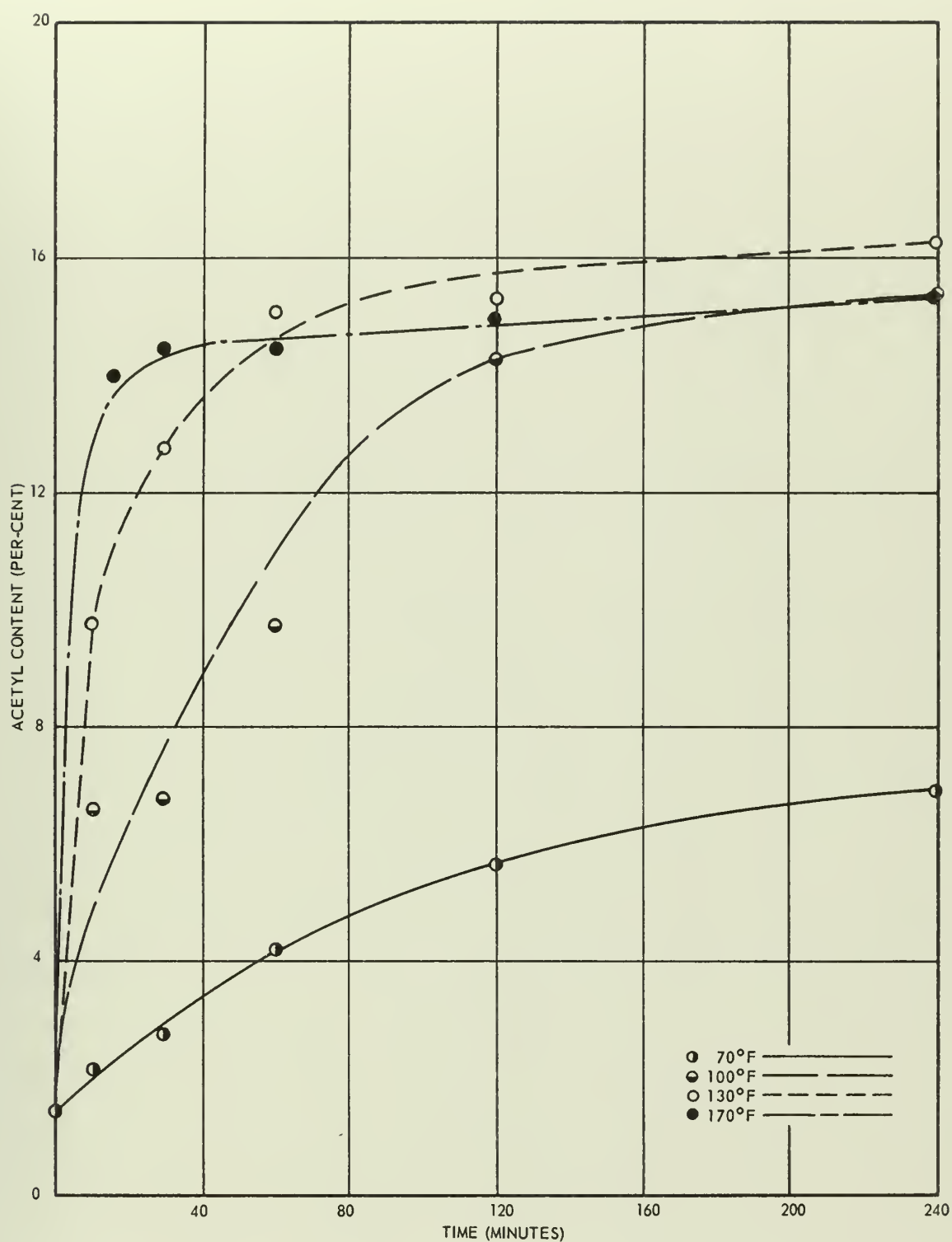


Figure 17. The Change in Acetyl Content with Presoaking Time for Acala 1517 Cotton at Different Presoak Temperatures.

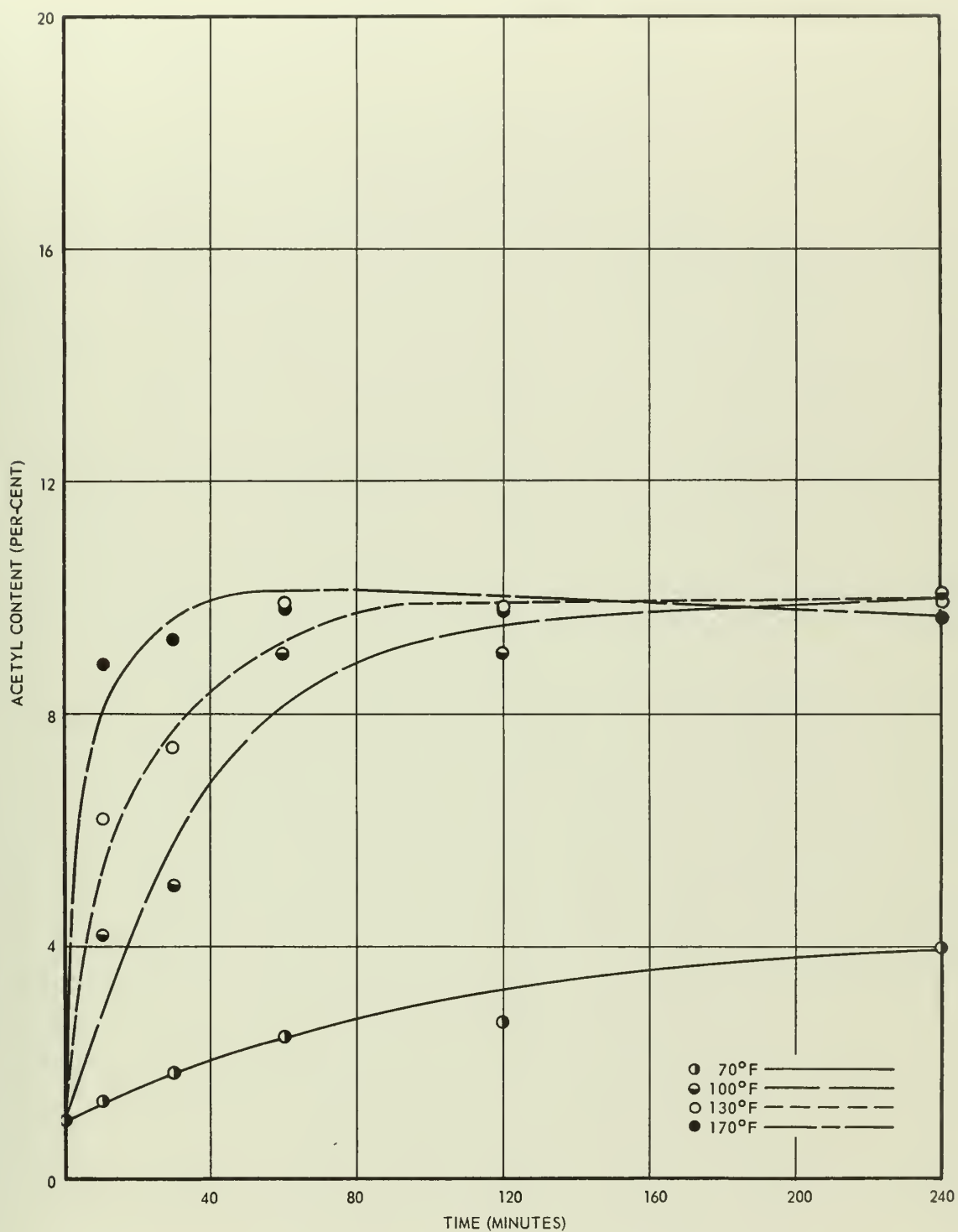


Figure 18. The Change in Acetyl Content with Presoaking Time for Lockett 140 Cotton at Different Presoak Temperatures.

CHAPTER VI

CONCLUSIONS

An increase in the temperature of the glacial acetic acid used for presoaking increases the rate in which maximum acetylation is reached under constant acetylation conditions.

Higher degrees of acetylation are obtained at the higher presoaking temperatures for short periods, 10 to 60 minutes, of presoaking.

Increasing the temperature of the presoaking acid increases the rate of acetylation but it does not effect the maximum degree of acetylation obtained under constant acetylation conditions.

The optimum time and temperature of presoaking (maximum acetylation in the shortest presoaking time) was found to be 10 minutes at 170° F.

Different fibers acetylated at different rates and degrees under constant conditions of presoaking and acetylation.

The order of maximum acetyl content under the same acetylating conditions obtained for the various fibers was Memphis, Acala 1517, Stoneville 2B, Empire Bale, Bob Shaw, and Lockett 140.

Generally the more immature the fiber the greater the rate and degree of acetylation under constant conditions of presoaking and acetylation and conversely the more mature the fiber the less the rate and degree of acetylation under constant conditions.

The higher the temperature of acetylation, the greater the degree of acetylation and the greater the degradation of the cotton fibers.

In general, under the same conditions of presoaking and time of acetylation, an increase of 24° F., from 58° F. to 82° F., approximately doubled the acetyl content obtained but weakened the fibers so that fiber strengths were unobtainable at 82° F.

The method of presoaking and acetylation used in this study produced evenly acetylated cotton fibers.

CHAPTER VII

RECOMMENDATIONS

It is recommended that further research be conducted into the effects of presoaking above 130° F. using different times and temperatures of acetylation. The conditions of time and temperature required for obtaining the desired acetyl content with the least amount of degradation could thus be determined.

The possibility of producing acetylated cotton on a continuous range by first scouring the raw stock followed by a short high temperature presoak and short acetylation should be investigated in order to insure uniformity in degree of acetylation of the various fibers, less degradation of the fibers and shorter overall time of production.

The use of high temperature presoaking in partially acetylating yarns and fabrics should be investigated.

The effect of other chemical and physical properties of the fibers, such as alpha cellulose, gum and wax content, ash content, and crystallinity, on the partial acetylation of cotton should be investigated.

BIBLIOGRAPHY

- A.S.T.M., "Tentative General Methods of Testing Cotton Fibers (No. D414-47T)," Standards on Textile Materials, October, 1948, p. 229.
- Blow, C. M. and Knight, W. J., "Textile Fibres and Rubber as Associated Materials for Manufacture," Textile Institute Journal Proceedings, 35 (1944), 12.
- Callow, H. J., "Acetylation of Jute," Journal of the Textile Institute, 43-8 (1952), T423-32.
- Chippindale, E., "Low Acetylation of Cellulose Fibres," Society of Dyers and Colourists, L (1934), 142-49.
- Copper, Albert S., and others, "Partial Acetylation of Cotton," Textile Industries, 116-1 (January, 1952), 97-102, 194-95.
- Doree, C., "The Action of Sea Water on Cotton and Other Textile Fibers," Biochemistry Journal, 14 (1920), 709-14.
- Elod, E., Schmid-Bielenberg, H. and Thoria, I., "Acetylation of Cellulose Fibers," Agnew Chemistry, 47 (1934), 465-68, as abstracted in Journal of the Textile Institute, 25 (1934), A420.
- Goldthwait, C. F., McLaren, J. and Voorhies, S. T., Jr., "Acetylated Cotton Highly Resistant to Rotting," Textile World, 96-1 (1946), 115-17, 212, 216.
- Goldthwait, Charles F., "Acetylation to Make a New Textile," Yearbook of Agriculture, 1950-51, 421-25.
- Heuser, Emil, "Factors Which Influence the Kinetics of Cellulose Reactions," Textile Research Journal, 20-12 (December, 1950), 829-36.
- Heuser, Emil, The Chemistry of Cellulose. New York: John Wiley and Sons, Inc., 1947, 660 pages.
- Heuser, Emil, and others, "Acetylation of Cellulose in Phosphoric Acid Solution," Industrial and Engineering Chemistry, 40 (1948), 1500-06.
- Hermans, Petrus, Physics and Chemistry of Cellulose Fibres. New York: Elsevier Publishing Co., Inc., 1949, 534 pages.
- Hermans, P. H. and Weidinger, A., "X-Ray Studies on the Crystallinity of Cellulose," Journal of Polymer Science, 4 (1949), 135-44.



- Honold, E., Poynot, J. and Cuculla, A., "Heat Resistance of Partially Acetylated Cotton Fabrics," Textile Research Journal, 22-1 (1952), 25-29.
- Howlett, F. and Martin, Elizabeth, "Acetylated Celluloses: Acetic Acid Yield; Determination," Journal of the Textile Institute, 35 (1944), T1-6.
- Kakurada, I., Tukakahara, I. and Morita, T., "Preparation of Fibrous Triacetylcellulose," J. Soc. Chem. Ind., Japan, 42 Supplemental binding (1939), 252, as abstracted in Chemical Abstracts, 34-1 1940, 619.
- Kanamaru, K., "Cellulose Acetate and Nitrocellulose: Refractive Index," Helv. Chim. Acta., 17 (1934), 1429-40, as abstracted in Journal of the Textile Institute, 26 (1935), A222.
- Knight, Henry G., "Cotton - Yesterday, Today and Tomorrow," Chemical Engineering News, 20 (1942), 581-83.
- Mackenzie, H.A.E., "Acetic Acid/Acetic Anhydride System: Properties," Transactions Faraday Society, 44 (1948), 159-71.
- Malm, C. J. and others, "Treatment of Cellulose Prior to Acetylation," Industrial and Engineering Chemistry, 44 (1952), 2904-09.
- Malm, C. J. and Clark, H. T., "The Action of Fatty Acids on Cellulose," Journal of the American Chemical Society, 51 (January, 1929), 274-77.
- Marsh, H., "Intermicellar Hole and Tube System in Fiber Structure," Journal of Physical Chemistry, 44 (June, 1940), 779.
- Mauersberger, H. R., Matthew's Textile Fibres. New York: John Wiley and Sons, Inc., 1947.
- Meller, Alexander, "Fibrous Cellulose: Reactivity," Journal of Polymer Science, 4 (1949), 619-28.
- Meyer, K. H., Natural and Synthetic High Polymers. New York: Interscience Publishing Co., Inc., 1950, pp. 387-393.
- Ott, E., Cellulose and Cellulose Derivatives. New York: Interscience Publishing Co., Inc., 1946, pp. 130, 607-20, 671-75.
- Petitpas, Genevieve, "The Perchloric Acid Catalyst of the Acetylation of Cellulose," Mem. Services Chim Etat., 31 (1944), 178-86, as abstracted in Chemical Abstracts, 40-2 (1946), 7613.

- Pettinger, A. H., "The Metallic Perchlorates," American Dyestuff Reporter, 27 (1938), 122-136.
- Scott, Walter M., "Some Recent Developments in Cotton Research at the Southern Regional Research Laboratory," Textile Research Journal, 19 (1949), 436-43.
- Sisson, W. A., "X-Ray Diffraction Behavior of Cellulose Derivatives," Industrial and Engineering Chemistry, 30 (May, 1938), 530-37.
- Skinkle, J. H., Textile Testing. Brooklyn, N. Y.: Chemical Publishing Co., Inc., 1949, 353 pages.
- Taniguchi, M., "Determination of Acetyl Value of Primary Cellulose Acetates," J. Soc. Chem. Ind. Japan, 44 (1941), 956-58, as abstracted in Textile Institute Journal, 40 (1949), A254.
- Ward, L., "Crystallinity of Cellulose and its Significance for the Fiber Properties," Textile Research Journal, 20-6 (1950), 363-72.



APPENDIX

Table 9. Per Cent Moisture Content of
Acetylated Samples Presoaked at 70° F.*

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	5.8	5.8	5.6	5.4	4.8	4.4
Empire Bale 92	5.4	5.7	5.7	5.6	5.4	5.1
Bob Shaw	5.4	5.5	5.5	5.4	5.3	4.9
Stoneville 2B	5.7	5.8	5.7	5.6	5.5	5.0
Acala 1517	5.7	5.8	5.8	5.9	5.3	5.3
Lockett 140	5.8	5.9	5.9	5.9	5.7	5.5

Table 10. Per Cent Moisture Content of
Acetylated Samples Presoaked at 100° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	5.8	4.9	4.7	4.4	4.0	4.0
Empire Bale 92	5.4	5.3	4.9	5.1	4.6	4.8
Bob Shaw	5.4	5.0	5.3	4.9	5.2	4.9
Stoneville 2B	5.7	5.4	5.1	5.0	4.2	3.9
Acala 1517	5.7	5.8	5.6	5.3	4.4	3.8
Lockett 140	5.8	5.2	5.3	5.5	5.1	4.4

*All acetylations in Tables 7-10, 12-15, and 17-20 were 45 minutes at 64° F.

Table 11. Per Cent Moisture Content of
Acetylated Samples Presoaked at 130° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	5.8	4.6	4.1	3.7	3.8	3.9
Empire Bale 92	5.4	4.8	4.7	4.4	4.0	4.2
Bob Shaw	5.4	5.2	4.9	4.5	4.4	4.3
Stoneville	5.7	4.8	4.5	4.2	4.2	4.1
Acala 1517	5.7	4.3	4.5	4.3	3.9	4.2
Lockett 140	5.8	5.2	5.0	4.5	4.5	4.4

Table 12. Per Cent Moisture Content of
Acetylated Samples Presoaked at 170° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	5.8	4.7	4.7	4.3	4.2	4.3
Empire Bale 92	5.4	5.2	5.1	4.6	4.9	4.5
Bob Shaw	5.4	5.4	5.0	4.9	4.2	4.4
Stoneville 2B	5.7	4.8	5.3	5.2	4.5	4.3
Acala 1517	5.7	5.0	5.3	5.0	4.6	4.3
Lockett 140	5.8	5.2	5.3	5.3	4.8	4.6

Table 13. Per Cent Moisture Content of
Samples Acetylated at Different Temperatures*

Cotton	58° F.	64° F.	70° F.	76° F.	82° F.
Memphis	4.9	4.7	4.0	4.2	3.9
Empire Bale 92	4.9	5.2	4.2	4.4	4.0
Bob Shaw	5.0	5.4	4.6	3.9	3.5
Stoneville 2B	5.3	4.8	4.3	4.2	3.7
Acala 1517	5.3	5.0	4.3	4.0	3.4
Lockett 140	5.4	5.2	4.3	4.4	3.7

*Acetylations in Tables 11, 16, 21, carried out for 45 minutes with a presoak of 170° F. for 10 minutes.

Table 14. Micronaire Readings (Fiber Fineness)
of Acetylated Samples Presoaked at 70° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	2.60	2.62	2.63	2.68	2.68	2.70
Empire Bale 92	3.95	3.87	4.00	3.90	3.90	3.97
Bob Shaw	5.43	5.30	5.37	5.32	5.35	5.40
Stoneville 2B	3.80	3.66	3.83	3.75	3.73	3.83
Acala 1517	4.27	4.20	4.30	4.22	4.25	4.28
Lockett 140	6.03	5.92	6.05	5.95	6.05	6.03

Table 15. Micronaire Readings (Fiber Fineness)
of Acetylated Samples Presoaked at 100° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	2.60	2.80	2.70	2.82	2.75	2.75
Empire Bale 92	3.95	3.90	3.78	4.10	3.87	3.92
Bob Shaw	5.43	5.35	5.07	5.35	5.27	5.43
Stoneville 2B	3.80	3.80	3.70	3.88	3.73	3.83
Acala 1517	4.27	4.30	4.17	4.30	4.23	4.35
Lockett 140	6.03	5.90	5.95	5.95	5.93	5.96

Table 16. Micronaire Readings (Fiber Fineness)
of Acetylated Samples Presoaked at 130° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	2.60	2.85	2.85	2.87	2.80	2.87
Empire Bale 92	3.95	4.02	4.00	4.13	3.95	4.01
Bob Shaw	5.43	5.47	5.40	5.50	5.22	5.42
Stoneville 2B	3.80	3.90	3.91	3.93	3.80	3.93
Acala 1517	4.27	4.35	4.33	4.45	4.25	4.30
Lockett 140	6.03	5.97	6.00	6.08	6.02	6.18

Table 17. Micronaire Readings (Fiber Fineness)
of Acetylated Samples Presoaked at 170° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	2.60	2.63	2.73	2.78	2.73	2.80
Empire Bale 92	3.95	3.93	4.08	4.00	4.10	4.17
Bob Shaw	5.43	5.38	5.40	5.43	5.45	5.40
Stoneville 2B	3.80	3.80	4.02	3.85	4.00	4.10
Acala 1517	4.27	4.22	4.42	4.35	4.35	4.52
Lockett 140	6.03	5.90	6.02	5.97	6.07	6.10

Table 18. Micronaire Readings (Fiber Fineness)
of Samples Acetylated at Different Temperatures

Cotton	58° F.	64° F.	70° F.	76° F.	82° F.
Memphis	2.68	2.63	3.00	3.08	3.18
Empire Bale 92	4.02	3.93	4.33	4.28	4.50
Bob Shaw	5.37	5.38	5.73	5.57	5.87
Stoneville 2B	3.65	3.80	4.08	4.10	4.23
Acala 1517	4.32	4.22	4.58	4.60	4.78
Lockett 140	6.00	5.90	6.27	6.10	6.18

Table 19. Pressley Index (Fiber Strength) of
Acetylated Samples Presoaked at 70° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	6.24	6.87	6.68	6.44	6.20	6.97
Empire Bale 92	6.99	7.16	6.92	6.69	6.64	6.51
Bob Shaw	8.11	8.20	8.13	7.93	8.07	8.02
Stoneville 2B	7.71	8.16	7.87	7.96	7.73	7.54
Acala 1517	8.63	8.32	8.11	8.11	8.04	8.01
Lockett 140	7.05	7.32	7.20	7.21	6.98	6.84

Table 20. Pressley Index (Fiber Strength) of
Acetylated Samples Presoaked at 100° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	6.24	6.84	6.37	6.10	5.91	5.70
Empire Bale 92	6.99	6.55	6.80	6.52	6.13	5.98
Bob Shaw	8.11	8.33	8.42	8.07	7.68	7.18
Stoneville 2B	7.71	7.13	7.09	6.93	6.68	5.99
Acala 1517	8.63	8.27	8.50	7.84	7.32	7.28
Lockett 140	7.05	7.43	6.96	6.99	6.85	6.48

Table 21. Pressley Index (Fiber Strength)
of Acetylated Samples Presoaked at 130° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	6.24	6.86	6.78	6.47	6.33	6.05
Empire Bale 92	6.99	6.90	6.39	6.38	6.27	5.91
Bob Shaw	8.11	8.08	7.82	7.74	7.01	6.87
Stoneville 2B	7.71	7.33	7.00	6.95	6.91	6.71
Acala 1517	8.63	8.19	7.64	7.58	7.14	7.04
Lockett 140	7.05	7.50	6.97	6.69	6.70	6.76

Table 22. Pressley Index (Fiber Strength)
of Acetylated Samples Presoaked at 170° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	6.24	6.89	6.38	6.16	5.97	5.49
Empire Bale 92	6.99	6.91	6.53	6.12	6.05	5.76
Bob Shaw	8.11	7.68	6.95	6.54	6.13	5.61
Stoneville 2B	7.71	6.64	6.21	5.72	5.98	5.88
Acala 1517	8.63	8.29	7.64	6.35	6.35	6.14
Lockett 140	7.05	7.25	6.56	5.97	5.83	5.40

Table 23. Pressley Index (Fiber Strength)
of Samples Acetylated at Different Temperatures

Cotton	58° F.	64° F.	70° F.	76° F.	82° F.*
Memphis	6.02	6.89	6.09	5.52	-
Empire Bale 92	6.62	6.91	6.49	6.16	-
Bob Shaw	7.70	6.68	6.84	6.33	-
Stoneville 2B	7.16	6.64	6.80	6.17	-
Acala 1517	8.01	8.29	6.93	6.83	-
Lockett 140	6.53	7.25	6.27	6.01	-

*The fibers were too weak to obtain a satisfactory break.

Thesis
096

Owens

A study of the effect of
time & temperature of pre-
soaking & the temperature
of acetylation ... of dif-
ferent varieties of cotton
fibers.

20763

Thesis
096

Owens

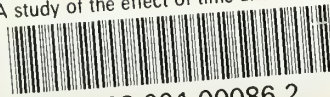
A study of the effects of pre-
soaking and the temperature of
acetylation on the rate and degree
of the partial acetylation of dif-
ferent varieties of cotton fibers.

20763

Library
U. S. Naval Postgraduate School
Monterey, California

thes096

A study of the effect of time and temper



3 2768 001 00086 2

DUDLEY KNOX LIBRARY